













# CHEMICAL ABSTRACTS

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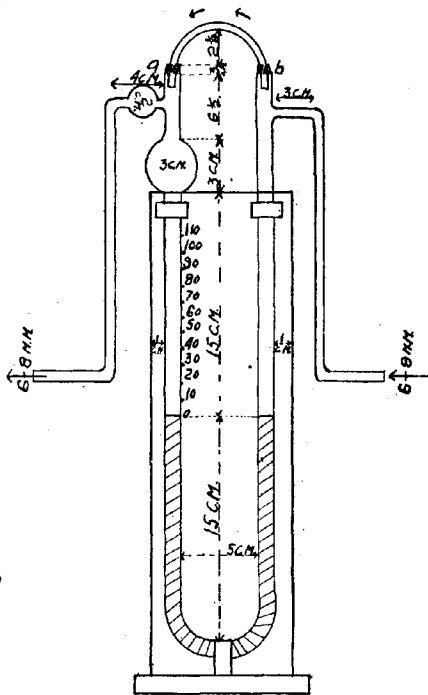
No. 1.

## I—APPARATUS

C. G. DERICK

Glass blowing. Internal welding. H. VIGREUX. *Chimie & industrie* 4, 334-6 (1920).—The difficulties of internal glass welding are discussed and directions for this operation are given. Cf. C. A. 14, 2845. E. H.

A new method for measuring rates of flow of gases. JOSEPH ERLICH. *Ann. chim. anal. chim. appl.* 2, 289-97 (1920).—App. for measuring rate of flow of gases is usually complicated, costly, and susceptible to corrosion. For higher rates, say 150 l. per hr., diaphragm meters are serviceable; for lower rates, up to 40 l. per hr., the method of counting bubbles may be utilized; for the range between these extremes the app. described appears to be admirably adapted. Poiseuille and Graham have shown that if a gas is forced through a capillary tube of diam. =  $d$ , length =  $l$ , and under a pressure  $h$ , the velocity at the outlet of the capillary,  $v$ , is given by the formula:  $v = Kh(d^4/l)$ , in which  $K$  is a const. depending upon the nature of the tube and gas. It is seen at once that  $v$  is a linear function of  $h$ , and this is shown to be true up to a certain value of  $v$  (70 to 100 l. per hr.). In the accompanying diagram, the capillary tube  $ab$  is attached at either end to the branches of a differential manometer. The manometer tube is filled half full of any suitable liquid, such as mineral oil or water satd. with the gas to be used. The gas should be under perfect control as it comes from the generator or container, preferably by use



of a 3-way stop cock, and it should pass in the direction indicated by the arrows. Before using, the app. must be calibrated. Choice of diam. and length for the capillary may be made by application of Poiseuille's formula, having given the data on one capillary

and a table of coeffs. of friction for various gases. Such tables are given in the original article, as well as a typical curve for air, in which  $v$  in l. per hr. is plotted as ordinate and  $h$  in arbitrary manometer scale readings as abscissa. A formula for correcting for errors due to change in temp. is also included.

J. T. R. ANDREWS

A new laboratory apparatus for mixing small quantities of a gas with other gases in constant proportions. ROBERT METZGER. Stuttgart. *Chem.-Ztg.* 44, 658-9 (1920).—Designed for mixing  $\text{NH}_3$  with illuminating gas, the app. is suitable for mixing other gases. It consists of a long-neck round flask standing in a Hg bath over a Bunsen burner with pilot flame. 3 parts moist  $\text{Ca}(\text{OH})_2$  and 1 part  $\text{NH}_4\text{Cl}$ , well mixed, are put in the flask which carries a stopper, and T-tube, 1 arm of which connects with an elementary analysis U-tube about half full of Hg. The arm of the U toward the flask has a ground stopper-cock; the other arm has a rubber stopper through which a gas-delivery tube with obliquely cut end extends nearly to the Hg. The nipple on this arm connects with the burner. The other arm of the T, through a 2nd T carrying a manometer, connects with a capillary tube which leads to a 3rd T, drawn to a nozzle, in the lower end of a large vertical mixing tube filled with glass wool. Through the other arm of this T the other gas is delivered to the mixer from a gas-holder. The rate at which the  $\text{NH}_3$  is evolved in the flask depends on the temp. of the Hg bath, which is closely regulated by the pressure on the Hg in the U-tube, this pressure being detd. by the rate of flow of the  $\text{NH}_3$  through the capillary tube to the mixer. J. H. MOORE

New automatic safety siphon. ANON. *Chem.-Ztg.* 44, 698(1920).—Made of strong glass, the long arm has a cock at the lower end and the short arm has a check valve. Between the arms at the top is fused a bell funnel with a long handle ground stopper at the bottom. To operate, with cock closed and funnel open the short arm is immersed in the liquid, the siphon then filled through the funnel, the stopper replaced in the funnel and the cock opened.

J. H. MOORE

Drying apparatus in which the material to be dried is moved by the heat carrier. B. WÄSSER. Magdeburg. *Z. angew. Chem.* 33, I, 229-31(1920).—Description, with 2 cuts, of the Bühler dryer in which the fine grained material is dried in 5-7 sec. by being blown by hot air or inert gases up a vertical pipe 45-60 ft. high to suitable cyclone separators. Cf. Bühler, *C. A.* 9, 1261.

J. H. MOORE

The theory of the katharometer. H. A. DAYNES AND G. A. SHAKESPEAR. *Proc. Roy. Soc. (London)* 97A, 273-86(1920).—This is largely a mathematical paper in which the corrections for and the theory of the app. designed by S. for automatically indicating the amt. of  $\text{H}_2$  in the air are considered by D. The elec. resistance of a thin Pt wire is influenced by its temp. which is increased in the presence of  $\text{H}_2$  (owing to surface combustion) in proportion to the amt. of  $\text{H}_2$  present. From the change in resistance of such a Pt wire can be calcd. the amt. of  $\text{H}_2$  in the gas surrounding it. In making this measurement a Wheatstone bridge was used in which two of the arms were manganin wire and the other two small helices of Pt wire (0.001 in. in diameter), enclosed in cells fitted in holes (1.5 cm. long by 0.6 cm. in diameter) drilled in a Cu block. In these cells each helix was mounted in a small frame consisting of a loop of Cu wire soldered to a Cu ring. Each helix was supported at its lower end by being soldered to the distal end of the loop, at its upper end to a Cu lead wire, which entered the cell through an ebonite plug. One of the cells was hermetically sealed while the other communicated to the atm. through 3 small holes. The resistance of each helix was about 8 ohms and the main working current of the bridge 0.1 amp., which gave the wires a temp. of about  $15^\circ$  above that of the block.

G. W. STRATTON

Bawtree colorimeter. W. GABLE. *Phot. J.* 60, 149-51(1919).—A long rectangular box is divided in halves lengthwise. Each part is lined with mirrors and a lamp at one end illuminates both. In the middle of one compartment a diaphragm of plain

glass and gelatin is placed; in the other is a color-filter composed of red, yellow and blue-violet in equal parts to give white light by color-mixture. Each diaphragm has a graduated shutter. The other end of the box carries two inclined opal reflectors and a device for viewing both simultaneously. A color filter at the diaphragm or a sample of pigment placed on the opal reflector can then be matched and measured in terms of the 3-color screens.

L. DERR

**Improved form of McLeod gage.** AUSTIN BAILEY. *Chem. News* 120, 302(1920).—In this modified McLeod gage the compression chamber is detachable, being connected to the main part of the app. by a ground-glass joint with a Hg seal. By the use of several compression chambers of different vols. but with capillary of the same size the app. may be used over a wide range of pressure.

G. W. STRATTON

**A thermoregulator.** J. FITCH KING. *J. Am. Chem. Soc.* 42, 2058(1920).—The oxidation of Hg at its point of contact with the adjustable wire in the ordinary Hg thermoregulator may be prevented by placing a few drops of Hg in a small bulb in the capillary a short distance above the point of contact and displacing the air in the space between this Hg and that which forms the contact with the wire by an inert gas. It was also found satisfactory not to displace the air in this space, when, after a short time of usage, the O<sub>2</sub> is all used up and the arc is then formed in an atmosphere of N<sub>2</sub>.

G. W. STRATTON

**The construction of thermocouples by electrodeposition.** WM. H. WILSON AND MISS T. D. EPPS. *Proc. Phys. Soc. London* 32, 328-40(1920).—If a wire of constantan, or other metal of high resistance, is plated with a sufficient thickness of Cu or Ag, the plated portion becomes a compound wire whose thermal e. m. f. against the unplated portion is nearly as great as for the pure Cu or Ag. A plating from 25 to 35% of the cross-section of the original wire is recommended. [The e. m. f. with constantan is then probably not over 14% less than with pure Ag and constantan. ABST.] By winding the original wire in a helix and plating one side of this, a series of plated and unplated pairs is obtained, forming a multiple-junction *thermel* (thermoelement). Very fine thermels can thus be made with very great ease. In one case a thermel of 4000 couples was made, whose hot end was less than  $1.6 \times 4$  cm.

W. P. WHITE

**New form of ozonizer for laboratory work.** Y. V. RAMAIAH AND M. V. N. SWAMY. *Chem. News* 121, 193(1920).—The app. is readily constructed from materials ordinarily found in the lab. and yields 40-60 mg. O<sub>3</sub> per l. O<sub>2</sub>. A method for the *estimation of ozone* has been developed, based on the work of Molinari and Socini (*Ber.* 39, 2735). Gas containing O<sub>3</sub> is bubbled through a 5-bulb absorption app. containing oleic acid, with CaCl<sub>2</sub> tube. The oleic acid quant. absorbs the O<sub>3</sub>, and the gain in wt. of the bulb and CaCl<sub>2</sub> tube corresponds to the wt. of O<sub>3</sub> in the gas.

DONALD W. MACARDLE

**An easily constructed check valve for water pumps.** ERWIN PINOFF. *Chem.-Ztg.* 44, 671(1920).—A piece of glass tube about 10 cm. long and 8 mm. inside diam. is carefully drawn down at 1 end to fit the suction hose. A piece of glass rod about 3 mm. diam. and 5 cm. long is heated at 1 end and pressed into a disk of such diam. that it will easily slip into the tube. The disk should not be perfectly round. A piece of rubber tubing 6 mm. long, 2 mm. inside and 6 mm. outside diam. is slipped on the rod nearly to the disk. Another piece of the same tubing, 2 cm. long, is slipped on the other end of the rod for a distance of 1 cm. and the whole is placed in the glass tube, the other end of which is then drawn down to fit the suction hose. In use, the tube is inserted in the line with the disk end of the rod toward the pump. Other dimensions may be used, care being taken that the rubber tubing fits tightly on the glass rod.

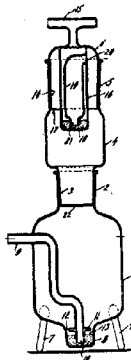
J. H. MOORE

Some new physical apparatus (BAWTREY) 26. Neon lamps for stroboscopic work (AUSTON) 4.

JORDAN, H.: Die Drehbare Trockentrommel für ununterbrochenen Betrieb. Leipzig: Otto Spamer. For review see *Chem. Ind.* 43, 432(1920).

Acetylene lamps. HARRY GALL. Can. 205,840, Nov. 23, 1920. Structural features of a lamp suitable for miners' use.

Laboratory absorption tube. W. R. FLEMING. U. S. 1,354,736, Oct. 5. The device is adapted for  $\text{CO}_2$  absorption in detg. C content of metals. In use, the space between the flange 11 and the body 1 is filled with moist cotton. Over this a small amt. of asbestos is placed, and the main portion of the chamber 1 is filled with an absorbent such as soda lime,  $\text{NaOH}$ ,  $\text{KOH}$  or a mixt. of caustic alkali and asbestos with a packing of asbestos just below the opening 22. A Hg seal closes the end of the tube 10. A packing of dry cotton is placed around the opening 20 and the remaining space of the chamber formed by the walls 16 and 14 is filled with dry asbestos. 27 indicates a Hg-seal. The openings 15 and 9 may be closed with a plug of dry cotton. The small bottom portion of the chamber 4 is filled with asbestos and the remainder of the chamber with  $\text{P}_2\text{O}_5$ .



Vacuum tubes. BRITISH THOMSON-HOUSTON CO. Brit. 147,822, July 9, 1920. In a process for removing ionizable gas from the anode of a vacuum tube, the anode is subjected to prolonged electron bombardment, preferably without appreciably heating it, the gas disengaged thereby being removed from the tube. For this purpose a voltage is applied between a filament cathode heated to incandescence and the anode of a value insufficient to produce a blue glow, the voltage being raised as the gas is removed; the final voltage is preferably higher than the working voltage unless this is very large, say 50,000 volts. Anodes so treated may be removed from the tube without re-absorbing gas.

Apparatus for cooling and crystallizing hot solutions. MASCHINENBAU-AKT. GES. BALCKE. Ger. 319,968, Aug. 10, 1918. Continuously running endless bands pass through the liquid to be cooled and carry the adhering hot soln. through a cooling air current. The crystallized salt is sepd. from the bands as the latter pass over rollers, the sepn. being facilitated by percussion or scraping. The crystals and liquid which fall from the bands are conducted to the liquid container which has a sloping bottom, and are later withdrawn from the lowest portion of the bottom.

Apparatus for treating liquids with liquids. B. DE M. DE VIVIES. Brit. 147,742, July 8, 1920. Relates to app. for washing liquids or for treating a liquid with a solvent immiscible with it and of different sp. gr. It is stated to be specially suitable for washing benzene, gasoline, etc., for extg. light constituents from gasoline, art oils, etc., by means of liquid  $\text{SO}_2$ , for sepg. tanning exts. and alkaloids from aq. soln., and for treating with alc. oils and fats that have been used for extg. perfumes.

Apparatus for producing liquid oxygen from air. C. F. CROMMETT and O. J. CROMMETT. U. S. 1,354,380, Sept. 28.

Apparatus for oxidizing gases. F. C. SCHMITZ. Can. 205,723, Nov. 16, 1920. The app. is for oxidizing gases by means of an opposed current of  $\text{O}$  and is specifically applied to the *manuf. of  $\text{P}_2\text{O}_5$* .

Apparatus for cleaning gases. FRANK R. MCGEE. Can. 206,155, Nov. 30, 1920.

Apparatus for separating oil and water or other liquids by sedimentation. J. BANNON and F. W. THOROLD. U. S. 1,355,934, Oct. 19.

Condensers and separators for compressed air. JAY G. McDOWELL. Can. 206,063, Nov. 30, 1920.

Depositing metallic films; electron discharge devices. BRITISH THOMSON-HOUSTON Co. Brit. 148,130, July 9, 1920. A gas-free film of metal such as W, Ta, or Mo is deposited on a glass, quartz, or other surface by vaporization of the metal in a vacuum at a pressure below 0.001 mm. Hg. A suitable construction is specified.

Catalytic heating apparatus. R. CAMELL, L. COCHER and J. GRITTE. U. S. 1,354,090, Sept. 28. The app. furnishes heat by the catalytic action of spongy Pt on alc. vapor or similarly acting fuel. Cf. C. A. 13, 2620.

Gas-calorimeter. W. G. LAIRD. U. S. 1,354,568, Oct. 5. Gas is burned within an absorption chamber through which H<sub>2</sub>O may be passed to a supply tank from which the gas passes to the absorption chamber. The rise in temp. of the H<sub>2</sub>O passing through the absorption chamber is measured.

Evaporator for distilling sea water. G. H. BENJAMIN. U. S. 1,355,935, Oct. 19.

Funnel. I. PEDERSEN. Japan 35,868, Feb. 21, 1920. The stem of the funnel is equipped with spiral grooves.

## 2—GENERAL AND PHYSICAL CHEMISTRY

WILLIAM E. HENDERSON

D. E. Tsakalotos. A. E. TSAKALOTOS. *Chem.-Zig.* 44, 765(1920).—Obituary.

E. H.

The scientific teaching of science. C. G. MACARTHUR. *Science* 52, 347-51 (1920).

E. H.

Scientific literature and industrial research. P. DUNSHEATH. *Electrician* 85, 570(1920).—The importance is emphasized of properly filing and indexing scientific publications.

C. G. F.

Coördination in libraries and abstracts. A. B. EASON. *Electrician* 85, 471-2, (1920).—A plea for the better and faster dissemination of technical knowledge. Suggestions are given for the improvement of existing methods.

C. G. F.

The uniformities of nature. CHARLES EDWARD FAWSITT. Sydney. *Roy. Soc. N. S. Wales. Presidential Address* (Dec. 3, 1919).—Miscellaneous generalizations relating to chemistry and biology.

NORRIS F. HALL

Fluidities and specific volumes of mixtures of benzyl benzoate and benzene. XXIX. EUGENE C. BINGHAM AND LANDON A. SARVER. Lafayette College. *J. Am. Chem. Soc.* 42, 2011-22(1920).—Kendall and Munroe (*C. A.* 11, 2850) had found that the fluidities of mixts. of benzyl benzoate and benzene are not additive although the substances are apparently inert, and they proposed an empirical formula. The authors working over a range of temp. find that these mixts. are not entirely inert, there being a very noticeable contraction on mixing which may account for the deviation noticed by K. and M. The empirical formula of K. and M. holds for one temp. only.

EUGENE C. BINGHAM

An accurate method for the determination of vapor pressure. E. J. HARTUNG. *Trans. Faraday Soc.* 15, Pt. 3, 150-9(1920).—The differential tensimeter method has been developed to reduce the error of measurement to only 1/10 of that given by previous methods. The app. used is described in detail. It is a development of the usual tensimetric app. A satd. soln. of KCl containing much undissolved salt is used as the reference liquid and dibenzyltoluene is used as the manometer liquid because of ease of handling and low vapor pressure, about 35-40 mm. at 280°. The method of prepn. is given. The exptl. technic is carefully described and results are given for KCl, K<sub>2</sub>SO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub>.

E. H. DARBY

**Some properties of copper ferrocyanide.** E. J. HÄRTUNG. *Trans. Faraday Soc.* 15, Pt. 3, 160-77 (1920).—After a consideration of previous work on Cu ferrocyanide, largely from the point of its importance in osmotic pressure work, H. takes up the measurement of the vapor pressure, the equil. between moist Cu ferrocyanide and aq. vapor, and the absorption of KCl and  $K_2SO_4$  from soln. The vapor pressure measurements were made by the method described in a previous paper (cf. preceding abstr.). The equil. curve was obtained by the method of van Bemmelen, which consists in plotting % water in gel against vapor pressure, but the results obtained were not altogether conclusive. Absorption was measured by detn. of the concn. of the salt solns. used before and after they had been in contact with the Cu ferrocyanide for 2 days. The absorption of  $K_2SO_4$  was shown to be greater than that of KCl. Results are given in the form of tables and curves. Apparently impermeability of the gel is not conditioned by the non-absorption of the solute as held by Tinker, at least as far as these results go.

E. H. DARBY

**Solubility. IV. Solubility relations of naphthalene and iodine in the various solvents, including a method for evaluating solubility data.** JOEL H. HILDEBRAND AND CLARENCE A. JENES. *J. Am. Chem. Soc.* 42, 2180-9 (1920); cf. *C. A.* 13, 1663.—An outline is given of a method for criticizing soly. data, according to which the solubilities of a substance in different solvents, where no mol. changes are involved, when plotted as the log.  $N$  against  $1/T$  give a family of straight lines converging to the m. p. of the solute where  $N = 1$ . Here  $N$  is the mol. fraction of the solute in soln. The position of these lines corresponds to the internal pressures of the solvents and is approx. predictable from them. The method is illustrated by means of soly. data of  $C_{10}H_8$  in  $CHCl_3$ ,  $C_6H_5Cl$ ,  $C_6H_6$ ,  $CS_2$ ,  $CCl_4$ ,  $C_6H_5CH_3$  and  $C_6H_{14}$ , and by data obtained from measurements of the soly. of I in  $C_6H_6$ ,  $CCl_4$  and  $C_7H_{16}$  at different temps. The influence of solvation upon soly. and its temp. coeff. is outlined. Critical solution temperatures of white phosphorus with various liquids. JOEL H. HILDEBRAND AND THRO. F. BUNHERR. *Ibid.* 2213-8.—Predictions made in the preceding paper are confirmed by the following values which have been detd. for the critical temp. of mixing of liquid P with other liquids:  $C_{10}H_8 > 300$ ;  $C_6H_5Cl$ , 264;  $C_{10}H_8$ , 202; phenanthrene 200;  $p\text{-C}_6\text{H}_4\text{Br}_2$ , 163;  $CS_2$ , -6.5.

H. JERMAIN CREIGHTON

**Determination of the last of the three functions which define the equation of state of ether.** E. ARIÈS. *Compt. rend.* 171, 154-6 (1920).—The calcd. values of the vol. of liquid and vapor are compared with the exptl. results of S. Young and the results are satisfactory. The form of equation and methods have previously been presented (*C. A.* 14, 2569).

E. D. WILLIAMSON

**Isotherms in the neighborhood of the critical state—the adiabatic compressibility of saturated vapors.** C. RAVEAU. *Compt. rend.* 171, 235-8 (1920).—A mathematical discussion of approximation to the equations for isotherms.

E. D. WILLIAMSON

**The thermodynamic properties of fluids in the neighborhood of the critical state.** C. RAVEAU. *Compt. rend.* 171, 471-3 (1920).—R. gives a neat and direct proof of equations for the slope of adiabatics and isotherms, but no new theorems are enunciated.

E. D. WILLIAMSON

**Remarks on the compressibility of saturated vapors.** G. BRUHAT. *Compt. rend.* 171, 162-4 (1920).—Equations are deduced for the slope of the adiabatics at points on the satn. curve. These show that "the adiabatic compressibility of a saturated vapor  $\partial v / \partial p$  is always negative" and "in absolute value the adiabatic of a gas always makes a smaller angle with the pressure axis than does the adiabatic of the liquid-vapor mixture." The sp. heat of the satd. vapor enters into the equation and B. points out that this quantity is large and negative at low values, tending to minus infinity at zero abs.

E. D. WILLIAMSON

The specific heat of saturated vapors at low temperatures. E. ARIÈS. *Compt. rend.* 171, 456-69(1920).—A. combats the reasoning put forward by Bruhat (preceding abstract) that the sp. heat of a satd. vapor tends to minus infinity at zero abs. and holds to the view that the limit is zero. Two other propositions are established: (a) the heat of vaporization tends to zero; (b) the entropy of the vapor approaches the value for a perfect gas.

E. D. WILLIAMSON

Heat of oxidation of beryllium. H. COPAUX AND CH. PHILIPS. *Compt. rend.* 171, 630-2(1920).—The Be used was obtained by electrolysis of molten sodium fluoroberyllate, as described by Lebeau. After failing to obtain complete oxidation by the usual method in a bomb calorimeter, an indirect method, based upon the sep. detns. of the heats of soln. of the metal and its oxide in the same acid, HF, was found to be successful. The mean of several of their best results shows Be to have a heat of oxidation of 151.5 Cal., thus placing it immediately after Ca and just before La in a table of heats of oxidation of elements.

J. T. R. ANDREWS

The specific heat of ammonia. HENRY A. BABCOCK. *Proc. Am. Acad. Arts Sci.* 55, 327-409(1920).—By measuring the rise in temp. produced by a measured quantity of elec. energy in a specially devised calorimeter, the sp. ht. at the const. sp. vol. of 2.6593 cc./g. of a mixt. of the liquid and vapor phases of  $\text{NH}_3$  was detd. between 30° and 125°. A special method of computing calorimetric curves and cooling correction is developed, and equations are given which permit the computation of any other sp. ht. in the liquid-vapor region or its boundary from data on sp. ht. at const. sp. vol. Satn. sp. hts. of the liquid and vapor phases are computed as far as 100°. Some of the values are given.

T	$C_{p2.6593}$	$C_{s2}$	$C_{s1}$
30	5.075	4.888	-3.663
40	5.190	4.991	-3.578
50	5.313	5.111	-3.535
60	5.446	5.252	-3.550
70	5.587	5.428	-3.633
80	5.737	5.665	-3.798
90	5.907	5.986	-4.106
100	6.165	6.433	-4.676
105	6.344	.....	.....
110	6.570	.....	.....
115	6.878	.....	.....
120	7.262	.....	.....
125	7.802	.....	.....

Here T is degrees C.,  $C_{p2.6593}$  is the sp. ht. of a mixt. of liquid and vapor at the const. sp. vol. of 2.6593 cc./g.,  $C_{s2}$  is satn. sp. ht. of the liquid, and  $C_{s1}$  is satn. sp. ht. of vapor. The latter it will be noted are negative. The principle sources of error in the method are discussed.

A. E. STERN

Viscosity of gel-forming solutions. W. R. HESS. Univ. Zürich. *Kolloid-Z.* 27, 154-63(1920).—Values obtained by the different experimenters for the viscosity of blood do not agree and a similar discrepancy has occasionally come to light in other colloidal systems. The discordant results are discussed and an account is given of the experimental work performed in the author's lab. for the purpose of discovering the cause of the discrepancies. A special form of viscosimeter is described in which the liquid is sucked through a capillary tube by reducing the pressure at one end. In the case of water or water-glycerol soln. the time of flow multiplied by the reduction of pressure in mm. is equal to a const. This is not true under all conditions for many colloidal solns. such as starch or gelatin in water. The values obtained differ by as



much as 100%. For this reason viscosity measurements in colloidal systems should be made under definite conditions of head or reduced pressure. It was observed that a 1.5 % starch soln. when stirred by a circular motion in a vessel, did not come to rest immediately when the stirring was stopped. A spontaneous pendulum motion occurred in the liquid which did not cease until several oscillations had taken place. According to the author these oscillations and the irregular viscosity measurements may be attributed to the fact that, in gel-forming solns. certain forces are at work tending toward the establishment of a definite structure. Because of the presence of these forces viscosity measurements in colloidal solns. do not always give true values for the internal friction. As a consequence these terms should not be considered synonymous. E. B. SPEAR

The application of Smoluchowski's theory to the movement of the particles of an emulsion. P. C. v. ARKEL. *Physik. Z.* 21, 465-7(1920).—A. considers Smoluchowski's equation  $\Delta n = (\nu - \pi)P$  where  $P$  is the probability of the expulsion of a particle that for the time 0 lies in the element taken.  $\nu$  is the number of particles which at equal states of division will be pptd. out of the vol. in question. It does not give results in accord with the exptl. data. A number of tables is given in support of this contention.

E. H. DARBY

Gelatin as an emulsifying agent. H. N. HOLMES AND W. C. CHILD. Oberlin College. *J. Am. Chem. Soc.* 42, 2049-56(1920).—Measurements of surface tension, viscosity, stability on long standing and concn. of gelatin in the 2 layers were made on kerosene-water emulsions containing varying amts. of gelatin. The effect of liquefying salts, such as the iodides and thiocyanates of the alkali metals, and of solidifying salts such as sodium tartrate, citrate and sulfate, was also studied. The authors conclude: First, that in order to obtain the most stable emulsions the max. lowering of the surface should be obtained, the viscosity should not be raised much beyond that of water and that an excess of acid, base or liquefying salt should not be employed. Second, there is no convincing evidence that gelatin is withdrawn from the soln. to form a layer around the oil droplets. Third, both viscosity and the lowering of surface tension are factors but the former plays the predominant role. E. B. SPEAR

Determination of the hydrogen-ion concentration in pure water by a method for measuring the electromotive force of concentration cells of high internal resistance. H. T. BRANS AND E. T. OAKES. Columbia Univ. *J. Am. Chem. Soc.* 42, 2116-31 (1920).—The H-ion concn. of pure  $H_2O$  has been detd. by measuring the e. m. f. of the concn. cell,  $Hg|HgCl.KCl|KCl|pure\ H_2O|H_2$ . To overcome the difficulty of measuring the e. m. f. of cells of this type, owing to the lack of sufficient amperage to actuate the galvanometer, a sufficient quantity of elec. is withdrawn from the cell, sorted up in a condenser of adequate capacity, and then discharged instantaneously through the galvanometer. By this method the e. m. f. of these cells may be detd. with an accuracy of 0.5 milli-v. The value obtained for the H-ion concn. of pure  $H_2O$  is  $1.23 \times 10^{-7}$ .

H. JERMAIN CREIGHTON

The polarity of an aluminium cell. FRANZ STREINTZ. *Graz. Physik. Z.* 21, 477-80(1920); cf. *C. A.* 14, 1249.—The cell,  $Al|H_2SO_4|Pt|H_2SO_4|Al$ , has been studied to det. whether the processes occurring therein are analogous to the unipolar conductance in crystals. The current polarity,  $U$ , is expressed by the equation:  $U = (I_{11} - I_{12})/I_{11}$ , where  $I_{11}$  is the current strength if the Al is cathode and  $I_{12}$  the current strength if the Al is anode. It has been found that  $U$  increases with the current strength, and that the following relation exists between the current strengths,  $I_{11}$  and  $I_{12}$ :  $I_{12} = -a/2\sqrt{(a/2)^2 + bI_{11}}$ , where  $a$  and  $b$  are coeffs. which increase with the voltage ( $V$ ). The variation with voltage of the "polarity effect" ( $UV$ ), for  $I_{11} = 0.01$  Weber, is similar to that found for argentite, and from 120 v. upwards  $UV$  is approx. const. With increasing current strength ( $I_{11}$ ) the voltage at which  $UV$  first becomes const. decreases.

H. JERMAIN CREIGHTON

Study of transition points by a dilatometric method. PAUL BRAESCO. *Ann. phys.* [9] 14, 5-75(1920); cf. *C. A.* 14, 876.—By a modification of Chevenard's differential method (*Rev. metal.* 15, (1917); cf. *C. A.* 11, 2743) giving the difference between the expansion of the sample and that of a standard substance, B. has detd. the temps. at which abrupt changes of vol. occur, the changes being due to changes of phase. For standard an alloy of Ni and Cr (10% Cr) was entirely suitable, as the expansion of the alloy shows no irregularities up to 1000° and as the transition point of Ni is much depressed (to -175°) by the 10% Cr. Many of the materials being powders were made into compact form by compression and by the addition of 5% of sodium silicate. The following results were found for  $\text{SiO}_2$ : amorphous silica by pptn. had no transition; quartz  $\alpha$  changes to quartz  $\beta$  at 575°; cristobalite  $\alpha$  to  $\beta$  at 235°; tridymite  $\alpha$  to  $\beta$  at 110-115° and to  $\gamma$  at 145°; quartz and amorphous silica to cristobalite above 1400°; tridymite to cristobalite at 1800°; quartz to tridymite between 1300° and 1400°. Thus cristobalite is the stable form above 1600°, while below 1600° tridymite is stable, and at low temps. quartz is the stable form. The mean coeff. of expansion of quartz is  $12 \times 10^{-6}$  between 100° and 300°;  $14 \times 10^{-6}$  between 200° and 300°; and  $21.3 \times 10^{-6}$  between 300° and 500°. A study of clays and clay constituents showed the following: The expansion of mica is regular up to 900°; natural clays show a decided break in their expansion at temps. from 630° to 840°, depending on the particular clay; kaolinite, however, showed a max. expansion at 500°, which confirms the work of Wallach (*C. A.* 7, 3847) who attributes the effect to dehydration of  $2\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ . Expts. with alloys of Cu and Sb varying from 4 to 100% Sb show that the 38.6% Sb alloy has the greatest coeff. of expansion. This compn. corresponds to  $\text{Cu}_5\text{Sb}$ , whose existence is claimed by LeChatelier (*Compt. rend.* 128, 1444) and is disputed by Kurnakov, Naberezhnov and Ivanov (*C. A.* 11, 3158). Samples of brass (Cu-Zn) from 37 to 60% Zn show abnormal expansion corresponding to the transition  $\beta \rightleftharpoons \alpha + \gamma$  of the diagram of Carpenter (*J. Inst. Metals* 5, 127 (1911)). Mechanical difficulties prevented the study of alloys richer in Zn. For the bronzes (Cu-Sn) the position of the transition  $\beta \rightleftharpoons \alpha + \delta$  found by Heycock and Neville (*Trans. Roy. Soc. London* 202, (1904)) at 500° has been confirmed for alloys between 14 and 29% Sn. Between 100° and 300° the coeff. of expansion is about  $20 \times 10^{-6}$ . Unfortunately the alloys do not possess sufficient mechanical resistance to permit these measurements above 550°. For the alloys of Cu and Al the temp. for the transition  $\beta \rightleftharpoons \alpha + \delta$ , 520°, confirms the results of Andrew (*C. A.* 9, 1597).

JAMES M. BELL

The influence of electrolytic dissociation on the distillation in steam of the volatile fatty acids. JOSEPH REILLY and W. J. HICKINBOTTOM. *Sci. Proc. Roy. Dublin Soc.* 16(N.S.), 120-30(1920).—In an earlier communication (*C. A.* 13, 2653) it was shown that the distn. const. of the lower satd. fatty acids, as calcd. from Nernst's distribution law, varied with the diln. This variation might be due to (a) variation in exptl. conditions, (b) impurities in the acids employed, (c) changes in the vols. of dil. solns. on further diln., or (d) deviation from the distribution law. Irregular condensation is prevented by jacketing the still-head with steam at 100°, and the other possibilities, except (d), are concluded to be negligible. Under the latter head, mol. aggregation is dismissed as improbable, but ionization would reduce the actual concn. of the undissociated acid and thus cause the calcd. values of the distn. const. to decrease with increasing diln. The Nernst expression is modified to admit the introduction of the dissoc. const., and a recalcn. of results on  $\text{HCOOH}$  and  $\text{AcOH}$  shows that the observed const. does not then vary with the diln. Study of the effect of added acids and salts confirmed the conclusion of Stein (*C. A.* 7, 3499) that the addition of  $\text{H}_2\text{SO}_4$  to aq.  $\text{AcOH}$  increases the distn. const.;  $\text{H}_3\text{PO}_4$  and  $\text{H}_2\text{C}_2\text{O}_4$  had a similar effect. The percentage change in the const. on addition of salts to the  $\text{AcOH}$  soln. was as follows:  $\text{NaOAc}$ , +5.2;  $\text{KCl}$ , +13.0;  $\text{KI}$ , +10.4;  $\text{NaCl}$ , +11.0;  $\text{K}_2\text{SO}_4$ , +10.3;  $\text{NaNO}_3$ , +10.6;

MgSO<sub>4</sub>, +13.2; ZnSO<sub>4</sub>, +9.3; SrCl<sub>2</sub>, +14.9; CuSO<sub>4</sub>, -5.1. No simple explanation of the results could be given.

W. B. V.

Applications of the method of distillation in steam. JOS. REILLY AND W. J. HICKINBOTTOM. *Sci. Proc. Roy. Dublin Soc.* 16 (N.S.), 131-42(1920); cf. preceding abstract.—Published data on steam-distn. of various compds. are collected and the regularities noted; a Ph group reduces the distn. const. while a OH group replacing COOH causes an increase; when both of the latter are present, however, the const. is generally less than in compds. containing only one of these radicals. Difference in constitution in isomeric substances affects the const. greatly, acids and alcohols containing a branched chain having higher consts. than normal ones. When associative effects are eliminated, it might be expected that the distn. const. would be proportional to the vapor pressure; in agreement with this view, the consts. of the fatty acids and alcs. reach a max. and then decrease with increasing mol. wt. A method for distinguishing the acids of butter-fat from those of other fats, depending on the presence of BuOH in the former, was successfully worked out; the filtered distillate from a Reichert-Meissl detn. was titrated with Ba(OH)<sub>2</sub>, the Ba pptd., and the soln. redistd. in a steam-jacketed flask. Successive fractions of the distillate were titrated. The acids of butter-fat gave a much lower percentage distg. than did other fats of high Reichert-Meissl number, such as babassu fat, coconut oil and palm-kernel oil. In the course of the work, other data of interest were obtained; vapor pressure at 100° of lauric acid, 0.058 mm.; of myristic acid, 0.033 mm.; distn. const.,  $\mu$ , of phenol, 1.7; *o*-cresol, 4.2; *m*-cresol, 3.15; *p*-cresol, 2.2.

W. B. V.

Electric figures. II. KARL PRZIBRAM. Vienna. *Physik. Z.* 21, 480-4(1920).—Elec. figures have been obtained by passing sparks from an induction coil between 2 metal plates placed upon a photographic plate. The variation of the range of the figures with increase in the length of the spark has been studied. It has been shown that the production of the negative discharge occurs before that of the positive. The magnitude of the figures formed in light dust has been detd. in air, N<sub>2</sub>, H<sub>2</sub>, O<sub>2</sub>, CO<sub>2</sub> and Cl<sub>2</sub>.

H. JERMAIN CREIGHTON

*p*-Cymene as a solvent. A. S. WHEELER. *J. Am. Chem. Soc.* 42, 1842-6(1920).—The price of *p*-cymene has decreased so much since it has been discovered to be the main constituent of spruce turpentine, a by-product of the manuf. of spruce pulp by the sulfite process, that it should be placed on the regular list of solvents; it is of importance as a solvent because it is a hydrocarbon of high b. p. (176.5°) and, where possible, is to be preferred to such colored solvents as PhNH<sub>2</sub> or PhNO<sub>2</sub> or an evil-smelling one like C<sub>6</sub>H<sub>5</sub>N. It should be useful as a simple solvent or in mixed solvents and in mol. wt. detns. by the b. p. method. In its purification the SO<sub>2</sub> is best removed by drawing air through it (10 hrs. for 5 gals.); it is then distd. with superheated steam, the vapors being passed through 30% NaOH before they are admitted to the condenser, and the distillate is shaken with 5% NaOH and finally distd. over Na. For pure *p*-cymene  $n_D^{20}$  is 1.4905. Below are given the solubilities in g. per 100 g. cymene of various substances: AcNHPh, 7.23 at 30°; anthracene, 1.57, 25°; 1.72, 30°; 9.24, 100°; 98.60, 176°; antipyrine, 2.09, 30°; caffeine, 0.76, 25°; 1.10, 30°; 1.74, 100°; 15.58, 175°; *d*-camphoric acid, 1.53, 100°; C<sub>6</sub>Cl<sub>4</sub>, 2.93, 23.5°; 16.77, 100°; 106.95, 176°; C<sub>10</sub>H<sub>8</sub>, 14.22, 30°;  $\beta$ -C<sub>10</sub>H<sub>7</sub>NH<sub>2</sub>, 8.63, 40°; *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, 6.33, 30°; *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, 1.36, 30°; *o*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, 0.83, 25°; phloroglucinol, 0.12, 175°; strychnine, 0.71, 30°; 0.95, 100°; thymol, 113.85, 25°; (the following acids are all at 25°) BzOH, 6.500-6.545; succinic, 0.020-0.022; anthranilic, 0.740-0.747; citric, 0.032-0.036; picric, 2.536-2.618; phthalic, 0.024-0.028; salicylic, 0.975-0.989. Tartaric acid, thiourea and urea are insol. at room temp. and nearly insol. at 100°. When perfectly pure, cymene does not change color within 2 hrs. in moderate light, but when it contains impurities

(such as *p*-anisidine) it turns pale or deep yellow or red, depending on the degree of impurity and the time of action.

CHAS. A. ROULLER

**Lecture experiment on combustion.** A. PRICE. *Chem. News* 120, 149-50 (1920).—By holding a wire, horizontally, in the gas escaping from a Bunsen burner (made from a long wide glass tube) and by regulating the flow of the gas and the distance of the wire from the end of the tube it is possible, by carefully igniting the gas, to cause it to burn only above the wire. This phenomenon may also be shown by suspending in the gas a small rounded object such as a bullet, when the luminosity takes on a different form. The phenomenon is of the nature of a "gas cap" which the miner gets by testing for fire damp by means of a safety lamp and illustrates the combustion of the gas at a temp. below its actual "ignition point."

G. W. STRATTON

**The practical significance of Ostwald's color system.** ALFRED WIRTH. *Chem.-Ztg.* 44, 737-8 (1920); cf. *C. A.* 11, 1358; 14, 492.—Colors are identified with one of Ostwald's 680 norms by the use of an app. (description lacking) furnished by Unesma, Leipzig. W. gives examples of how the color norms may be matched by blending proper quantities of white, black, and "bright" colors.

F. A. WERTZ

**Novel method for demonstrating solubility of silver salts.** H. LÜPPO-CRAMER. *Phot. Rundschau* 57, 23-5 (1920).—Agar cultures of staphylococci are made in Petric dishes, and strips of filter paper, over which damp Ag salts are lightly brushed, are laid on the jelly surface. After 24 hrs. incubation, compds. of soly. between  $1.7$  and  $2.7 \times 10^{-4}$  show bacteria-free zones 14-19 mm. wide; with  $\text{Ag}_2\text{S}$  the growth persists up to the edge of the paper.

L. DERR

**Neon lamps for stroboscopic work (ASTON) 4.** A new form of standard cell (RODMAN, SPOONER) 4. Two common failures of the Clark standard cell (McKELVY, SHOEMAKER) 4. Measuring rates of flow of gases (ERLICH) 1. Sorption by cellulose and starch (SCHERINGA) 11A.

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### 3—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

GERALD L. WENDT

**Recent developments in atomic theory.** WALTER PETERS. *Z. angew. Chem.* 33, 1, 133-6 (1920).—A review of evidence for at. complexity.

G. L. WENDT

**The constitution and character of the chemical elements.** HAWKSWORTH COLLINS. *Chem. News* 121, 157-9 (1920); cf. *C. A.* 14, 1255, 2123, 2748.—The method previously used is applied to most of the elements above Mn in the periodic table. C. finds that

when the nearest whole integer is taken instead of the generally accepted value for the at. wts. that many of the elements differ from each other in at. wt. by 23 or some multiple of it. So he concludes that Na is a very important unit in the construction of elements. The "probability" of this being an accidental relationship is calcd. to be very small. The heavier atom will differ in valence by  $\approx 1$  for each Na added. The five main principles in the constitution and structure of the chemical elements. *Ibid* 121, 182-5.—The first 4 have been "fully demonstrated" (C. A. 14, 1255, 2123, 2748 and preceding abstract). The fifth principle states that the heavier elements are formed by a union of simpler elements and this is "proved" by a combination of mineralogical and chemical facts. There are 9 pairs of elements which differ in at. wt. by 90, the approx. value for Zr, which is found intimately associated with most of these elements. Not much change in at. vol. is produced by the addition of Zr; e. g., Au = ZrAg. Similar evidence is offered supporting the proposition that Na is a constituent of most heavy metals; e. g., Cu = NaCa. F. O. A.

High frequency spectra of lead isotopes. C. D. COOKSEY AND D. COOKSEY. Yale Univ. *Phys. Rev.* 16, 327-36(1920).—By photographing on the same plate the upper half of the  $L_{\alpha 1}$  line from one kind of Pb, and the lower half of the same line from another, the authors compared the wave lengths of the lines. Ordinary Pb and Pb from a primary Indian uraninite believed to be free from ordinary Pb and nearly free from thorio-lead were compared, and no difference of wave length was found as great as 0.00006 Å or 0.005% of the wave length of the line studied. This result confirms and extends Siegbahn and Stenström's conclusion. (cf. C. A. 12, 19). NORRIS F. HALL

The independent origin of actinium. ELLIOTT QUINCY ADAMS. Washington. *J. Am. Chem. Soc.* 42, 2205-8(1920).—Piccard's hypothesis (cf. C. A. 12, 17) that the Act series springs from a 3rd isotope of  $U_I$  and  $U_{II}$  is shown not to involve identity of the periods of  $U_I$  and the 3rd isotope. A. assumes that P.'s Act-U is formed from a hypothetical  $\beta$ -radiating isotope of Protoactinium, which has long since all decayed. The at. wt. 235 is assigned to this atom-species, which gives Act a wt. of 227 and Act-Pb a wt. of 207, in agreement with Hönigschmid's detn. of 206.05 as the at. wt. of U-Pb containing approx. 3% of Act-Pb. The names "radon, thoron, actinon" are suggested for the emanations. NORRIS F. HALL

Radium. ROBERT M. KEENEY. *Mineral Ind.* 28, 622-3(1919).—Production, technology and uses are discussed. A. B.

Colloid chemistry and radium investigations. F. SEKERA. Vienna. *Kolloid-Z.* 27, 145-54(1920).—A résumé is given of the work recorded in the literature on the colloidal nature of radioactive substances. The bearing of colloidal chem. methods on radio-chemistry is discussed and the limitations are pointed out. The article is divided into the following sections: (1) Formation and destruction of colloidal dispersions by Ra emanation; (2) colloidal nature of radioactive substances; (3) adsorption phenomena in radio-chemistry; (4) colloid chemistry of fluorescence. E. B. SPEAR

The variation of the factor  $h$  in the equation of  $\frac{1}{2}mv^2 = h\nu$ . FERNANDO SANFORD. *Phys. Rev.* 16, 337-48(1920).—S. has shown elsewhere (C. A. 13, 1558, 2484) that by combining the equation for orbital motion of an electron about a central positive charge, viz.,  $(mv^2)/R = (Qe)/R^2$ , with the equation  $\frac{1}{2}mv^2 = h\nu$  it is possible to compute the central positive charge of a radiating atom from an equation which reduces to  $Q = (2.882 \times 10^{-12})/\sqrt{\lambda}$ . Using the wave length measurements of the K-radiation, made by Blake and Duane (C. A. 12, 649) the values of  $Q$  calcd. from the above equation were found to be equal to the atomic charges of the same elements calcd. from the Moseley formula by the equation  $Q = 2e(N-3.6)$ , where  $N$  is the at. number. By combining these two equations for  $Q$  it was possible to get an expression for the value of the wave length of the characteristic K-radiation which was found to apply to the elements from  $N = 35$  to  $N = 58$  within the limits of exptl. error (cf. C. A. 14, 1929).

At the same time it was shown that if the mass of an electron varies as its velocity varies, its rate of emission must also vary, or else the Moseley equation does not apply to the charges calcd. on the assumption of orbital motion. That the Moseley equation does apply so completely to the charges calcd. on the assumption that  $h^2/m$  remains const. is an argument against the hypothesis that  $h$  is const. Recently Duane and his colleagues (C. A. 14, 1259) have extended the wave length measurements on the absorption frequencies of the K-radiation to both lower and higher at. numbers, while Siegbahn and Jönsson (C. A. 14, 3191) have published new detns. of the wave lengths of highest frequency in the K-radiation series of a considerable number of elements from Cd to U. When these newly detd. wave lengths are computed by S.'s formula it is found that, for elements with an at. number greater than 58, the calcd. values are greater than the observed. Assuming only that  $Q$  increases by  $2e$  in passing from any element to the one of next higher at. number, it follows that the variation must be due to the fraction  $h^2/m$ . S. next plots the calcd. values of  $h^2/m$  as ordinates against their corresponding wave lengths as abscissas and finds that a smooth curve is obtained from which values of  $h^2/m$  may be taken for any wave length down to  $\lambda = 0.1 \times 10^{-8}$ . If corresponding values of  $v$  could be found it would be possible to calc. the respective values of  $h$ . Now all the calcns. made are based on the assumption that the speeds of electrons expelled under the stimulus of ultra-violet light or of X-rays are the orbital speeds which they possessed at the instant of breaking away from their respective atoms. If this be a correct assumption, it seems almost certain that the same explanation must account for the speeds of the  $\beta$ -particles expelled by radioactive bodies. This would require that the Einstein photoelec. equation,  $\frac{1}{2}mv^2 = h\nu$ , should hold for the energies of the  $\beta$ -rays and the frequencies of the accompanying  $\gamma$ -radiations from the same elements. Using the measurements made by Rutherford on the velocities of  $\beta$ -rays expelled from Ra B and from Ra C and by Rutherford and Andrade on the wave lengths of lines in the  $\gamma$ -ray spectrum of the same 2 elements, S. concludes that the following linear relation holds:  $h = 4.25 \log m + 2.50 \times 10^{-27}$ . From this relation and the equation giving the mass of an electron in terms of its own velocity and that of light, values of  $v$  from  $0.1 \times 10^{10}$  to  $40 \times 10^{10}$  were plotted against the values of  $v$ . From this curve it was possible to obtain a value of  $v$  accurate to the third significant figure. Values of  $v$ , corresponding to all the K-radiation frequencies measured by the investigators referred to above, were then taken from this curve and plotted against their respective at. numbers. The velocities calcd. in this manner bear a linear relation to the at. numbers with no tendency to systematic departure on either side of the line for higher at. numbers. It is concluded that since  $h$  may be replaced in Einstein's photoelec. equation by a function of  $m$  with a great improvement in its accuracy, there is no longer ground for speaking of an energy quantum in this equation.

A. L. FIELD

A study of the residual ionization in a gas with reference to temperature effects. C. H. KUNSMAN. Univ. Cal. Phys. Rev. 16, 349-61 (1920).—A form of electrometer was constructed which used the principle of the Wulf electrometer in sepg. the conduction of charge across the insulation from the conduction in the gas due to ionization. The new features of this apparatus were that it was possible (1) to make a quick and accurate test of the insulation properties under the same conditions as those under which the ionization tests were made, and (2) to subject the instrument to a wide range of temps. The exptl. data show that the number of ions generated per cc. per sec. within an air-tight chamber is 8.22 in the basement of the Physical Lab., Univ. of Cal., and 4.15 over the Pacific Ocean. The results of tests between  $-44.6^\circ$  and  $92.5^\circ$  show no indication that the residual ionization is due in any part to a mol. impact of thermal agitation. Observations made on the effect of changes in temp. on the insulation system would seem to account for the apparent daily and seasonal variations of the

residual ionization as previously reported. At temps. from  $-30.5^{\circ}$  to  $-44.6^{\circ}$  an increase in conduction across the insulation system was noticed, which was comparable to the apparent change as reported at high altitudes. It would therefore seem that it is not necessary to assume, as has been done by previous observers, that there is a highly radioactive layer in the upper atm., or that the sun is a source of penetrating radiation sufficient to generate 90 ions per cc. per sec.

A. I. FIELD

**The minimum arcing voltage in helium.** K. T. COMPTON, E. G. LILLY AND P. S. OLMSTED. Princeton Univ. *Phys. Rev.* 16, 282-9(1920).—Recent investigations (cf. Horton and Davies, *C. A.* 13, 1669; Franck and Knipping, *C. A.* 14, 2126) have shown that the minimum radiating potential of He is close to 20.2 v. and its minimum ionizing potential is 25.5 v. One of the authors (Compton, *Phil. Mag.*, in print) has shown that these values apply to radiation and ionization set up by a single electron impact against a normal unexcited atom, whereas, if the electron current and gas d. are relatively large, ionization may occur at any voltage above 20.2 v. The present expts. were made to det. the voltage at which the arc strikes under various conditions of gas pressure and electron current d. in very pure He. It was found that the arc could be made to strike at voltages as low as 20 v. but never lower. Under favorable conditions, however, the arc could be maintained at much lower voltages, the lowest voltage observed being 8 v., with a gas pressure of 5 mm. and a current of about 1 amp. through the gas. Observations of the spectrum of He were also made under various conditions. Apparently the ordinary He and *parhelium series lines* and the bands are excited whenever the arc strikes. The line  $\lambda 4686$  of the enhanced system was never observed below 55 v., and was stronger above 80 v. The lines of the sharp subordinate series of pairs are peculiar in that their intensity decreases, relatively to that of the rest of the spectrum, as the voltage is increased. The results are in accord with Bohr's theory of radiation and atomic structure. The fact that the lines of the so-called *parhelium series* appear at the same voltages as those of the He series renders untenable Stark's conclusion that they are due to atoms which have lost more electrons than those atoms which give rise to the He series lines.

A. I. FIELD

**Resonance potentials and low-voltage arcs for metals of the second group of the periodic table.** F. L. MOHLER, PAUL D. FOOTE AND W. F. MEGGERS. *J. Optical Soc. Am.* 4, 364-70(1920).—Previous publications discussing resonance and ionization potential measurements for five metals (Hg, Cd, Zn, Mg, and Ca) of the second group of the periodic table showed in every case a resonance potential corresponding by the quantum relation  $Ve = h\nu$  to the combination series line  $1.5 S - 2p_2$  (Paschen notation) and an ionization potential corresponding to the limiting frequency of this series,  $1.5 S$ . Ca showed, in addition, a second resonance potential corresponding to the frequency  $2.5 S - 2 P$ , the first line of the principal series of single lines converging to the same frequency,  $1.5 S$ . Spectroscopic studies of low-voltage arcs in these metals are not entirely in accord with the work on current-potential measurements, *e. g.*, the Mg line  $1.5 S - 2 p_2$  was not observed below the ionization potential. The presence of two resonance potentials for metals other than Ca, and the single line spectrum  $\lambda 4571$  for Mg are established by work reported in this paper. Computed and observed values of one ionization potential and of two resonance potentials are given for Zn, Cd, Hg, Mg, and Ca and the values to be expected for Sr and Ba. The following general laws for the phenomena of electron impacts with mols. of metals of the second group are stated: There are at least 3 types of inelastic impact, 2 resonance potentials and an ionization potential. (1) Electrons with kinetic energy exceeding one quantum of the frequency of the combination line  $1.5 S - 2 p_2$  may lose this amt. of energy in exciting radiation of this single frequency. (2) Likewise a quantum of the frequency of the first line of the principal series of sine lines  $1.5 S - 2 P$  may be transferred into radiation of this frequency. (3) Electrons with energy greater than that corresponding

to the common limit of the two absorption series, 1.5  $S$ , may ionize the mol. The many-line spectrum emitted above the ionization potential is probably to be ascribed to recombination of ionized atoms with electrons.

W. F. MEGGERS

Formation of the X-ray series with increasing atomic number. W. KOSSEL. *Z. Physik* 2, 470-8(1920).—K. shows there is some spectroscopic evidence for the electrons about the nucleus arranging themselves in shells of eight. Plots of  $\sqrt{\nu}$  against atomic number for the principal lines of the K, L, and M series show deviations from linearity until the shell immediately after the one concerned in their production is filled up.

F. C. HOYT

Law of reflection of X-rays. P. P. EWALD. *Z. Physik* 2, 332-42(1920).—The deviations from the Bragg formula  $n\lambda = 2d \sin \theta$  observed by Hjalmar (C. A. 14, 2750) can be partially accounted for by E.'s theory of the mechanism of reflection (C. A. 13, 1556).

F. C. HOYT

Spectrometric researches on X-rays. A. DAUVILLIER. *Ann. phys.* 13, 49-134 (1920).—D. has previously studied indirectly the cathodic spectra of discharges which take place in gas tubes and those of pure electron emission using the Blondel oscillograph to record the waves of potential and current which produce pulsatory, sinusoidal and const. e. m. f. (C. A. 11, 2996). The spectra of the independent and characteristic X-radiation corresponding to each of these cathodic spectra have been observed on a Bragg spectrometer, and important relations have been found to exist between them, which recall those found by Rutherford between the  $\gamma$  and  $\beta$  rays. The existence of the J absorption, discovered by Barkla, has been verified; the non-existence of the J radiation for the non-radioelements has been established. This fact was recognized by Duane and Tokéo Shimizu for Al. This is very important from the point of view of atomic structure and the origin of discontinuous spectra of high frequency. The oscillographic study of the discharge in the Coolidge tube has permitted the explanation of certain apparently anomalous phenomena. A comparison of the oscillographic and spectrometric studies of gas tubes and Coolidge tubes has brought to light their intimate properties and permits their precise comparison. The foundation has been laid for the precise spectrometry of X-rays together with the errors inherent in the method and the method of correcting them. A complete theoretical solution of the problem has been given and also an approx. exptl. one. A study has been made on the law of displacement of the max. of the spectra as a function of the velocity of the electrons. The law found is analogous to that of Wien relating to thermal radiation spectra, the potential playing the role of the abs. temp. The relation of J. J. Thomson, expressing the total energy of X-rays radiated as a function of the velocity of the electrons, has been verified. The results of the present work permit the standardization of X-rays, thanks to the Coolidge tube. D. proposes to replace the empirical and necessarily unprecise process of dosage in ordinary use by simple, direct measurements on the potential, current, distance and time of exposure. The const. fundamentals as set forth are (1) nature of the tube (pure electron emission), (2) nature of anticathode (tungsten), (3) the form of the e. m. f. (sinusoidal or better const.). The independent variables are (4) the max. voltage, (5) the mean intensity of current, (6) the nature and thickness of the filter interposed, (7) the distance from the target to the object, and (8) the length of exposure under const. conditions. These 8 conditions completely characterize the radiation and permit its rigorous reproduction, independently of any spectrometric measurements. The effect of electronic density on the target has been studied, both theoretically and experimentally. This study has suggested the construction, on new principles, of a generator of X-rays of great power and high efficiency. The conclusions of this work are in direct contradiction to the results of Lilienfeld, whose results have been explained. The emission of independent X-rays by the quanta was verified in 1916 between 24 and 109 kv. for gas tubes and incan-



descent cathodes and different anticathodic elements (W, Pt, Ni). There is offered a new absolute method of detn., by the spectra, of explosive potentials. An application has been made in air.

F. P. PHELPS

The law of mass absorption of X-rays. F. BUTAVAND. *Ann. phys.* 13, 161-8 (1920).—The absorptions by different elements of X-rays emitted by the different substances are grouped into 4 recognized series. The same element may give place to three series at most, and the same absorbing body two at most as nearly as can be judged from the known facts. In each series the coeff. of absorption  $\lambda/\rho$ , and the at. wts.  $P_a$  and  $P_e$  of the absorbing and emitting bodies are connected by the relation  $(\lambda/\rho)^{1/4} \times P_a/P_e = \text{Const.}$  The const. varies according to the series, increasing rapidly as the rays become softer. The value of the const. is given by the expression  $13 + 2^2(n-1)$ . The only large discrepancy is for the K series where the departure is 11 percent. The most general formulation becomes  $(\lambda/\rho)^{1/4} \times P_a/P_e^{1/2} = 13 + 2^2(n-1)$ . A very instructive chart has been drawn, plotting as abscissas the atomic numbers of absorbing bodies and as ordinates those of emitting bodies, from which it is deduced that there may be two additional series which have not yet been discovered, making in all six series, G, H, J, K, L, M, the extremes G and M being, one very hard and one very soft.

F. P. PHELPS

The K absorption bands of the rare earths for X-rays. M. DE BROGLIE. *Compt. rend.* 170, 725(1920).—The X-ray spectra of the rare earth elements have been useful in the classification and in fixing the position of those substances in series of the elements. B. has been able to obtain the K bands of the elements number 69, 70, and 71. With a sufficiently high dispersion the band of lutecium ( $N = 71$ ) can be seen very clearly between the two groups  $K\alpha$  and  $K\beta$  of W. The author has not attempted to make precise measurements but only to recognize without ambiguity the bands and the order of the elements. The K band of thulium ( $N = 69$ ) is at  $\lambda = 0.208 \times 10^{-8}$  cm.; of néo-ytterbium ( $N = 70$ ) at  $\lambda = 0.2015 \times 10^{-8}$  cm.; of lutecium ( $N = 71$ ) at  $\lambda = 0.195 \times 10^{-8}$  cm. The sample of thulium which contains néo-ytterbium shows very clearly the presence of both elements.

F. P. PHELPS

A new arrangement of crystal powder for X-ray crystallographic investigations. H. BOHLN. Tech. Univ., Danzig-Langfuhr. *Ann. Physik* 61, 421-39(1920).—A new arrangement is worked out for securing sharpness in the edges of interference lines with a greatly increased accuracy. The crystal powder is pressed into a curved cylinder having the same radius as the cylindrical camera. H. Seeman (*C. A.* 14, 1632) has developed a similar method independently. The sharpness and position of the edges of the lines are independent of the slit width and of the depth of penetration of the radiation. Observations made on Th and Ni indicate that they are composed of a lattice of face centered cubes of unit edge lengths of  $5.12$  and  $3.53 \times 10^{-8}$  cm. resp. Mg is built of two simple hexagonal lattices inserted into each other where  $a = b = 3.23 \times 10^{-8}$  and  $c = 5.25 \times 10^{-8}$ . The ratio  $a/c = 1/1.624$  whereas Hlawatsch (*Z. Kryst. Mineralog.* 32, 479(1900)) found the ratio  $1/1.625$ .

F. O. A.

The mechanism of chemical reactions promoted by X-rays. A. DAUVILLIER. *Compt. rend.* 171, 627-9(1920).—Since X-rays promote reactions through intermediate ionization it is suggested that the coloring action of these rays on various crystals and glasses is caused by the addition of electrons to the positively charged metallic ions so that the elements may be set free or their valence partially reduced; e. g.,  $\text{Cu}^{++}$  to  $\text{Cu}^+$  in silicate. Such free elements would then be capable of producing the photoelectric and thermoluminescent effects noted after X-rays had been allowed to act on crystals or glasses.

F. O. A.

The valency of photo-electrons and the photoelectric properties of some insulators. M. J. KELLY. *Phys. Rev.* 16, 260-73(1920).—Derieux (*C. A.* 12, 1725) in his work on the photoelec. effects on Hg droplets observed in a number of cases that the change in

speed of the droplets falling between the plates of a condenser while subjected to the radiation from a Hg lamp corresponded to the emission of more than 1 electron. If the changes which correspond to emission of more than 1 electron are due to simultaneous emissions from different atoms on the surface of the droplet, the chance of this occurring would be decreased by using less active surfaces and a lower radiation intensity. K. has extended the investigation to include several insulators. The apparatus was that used by Millikan in his evaluation of  $e$ . At the beginning of the expts. it was observed that the condenser plates emitted electrons so freely that the droplets captured about as many electrons as they emitted. A thin layer of paraffin applied to the plates stopped this emission completely and was retained in the subsequent measurements. When S droplets, atomized from liquid S at  $150^\circ$ , were illuminated with light from a Hg lamp passed through a fused quartz window 5 mm. thick, the emission rate was decreased to 6–8 per hr. About 200 emissions were observed at this reduced rate and in every case they were unmistakably attributable to single electrons. In the case of shellac the droplets were obtained by atomizing a filtered soln. of shellac flakes in EtOH. The alc. evaporates rapidly and the d. of the sphere reaches a const. value in a very few mins. The surface polarization or photo-emission fatigue observed in the case of S was absent here. The data obtained in 10 drops of shellac, corresponding to 303 emissions, disclosed nothing but changes due to single emissions. Expts. on paraffin droplets subjected to the action of radiation from the Hg lamp further corroborated the conclusions that the long-wave limit of paraffin is shorter than  $\lambda 2150$  (the shortest wave length photographed through the Hg lamp used). Under similar conditions no emissions were obtained with droplets prepared from the oil used by Millikan. However, using the radiation from a Zn spark passing through a thin fluorite window and about 12 cm. of air, *photoemissions* were obtained from both the *paraffin and oil* droplets. The long-wave limit of S was found to lie between  $\lambda 2400$  and  $\lambda 2200$ , while the same limit for shellac lies at some wave-length shorter than  $\lambda 2200$ . It is concluded that *photo-emissions* from mols. of S and shellac consist in the ejection of a single electron from the mol. at each emission.

A. L. FEND

Some general characteristics of spectro-photoelectrical conduction in solids. W. W. COBLENTZ. *J. Optical Soc. Am.* 4, 249–54 (1920).—Systematic search during the past 4 years has revealed various substances (molybdenite, thalofide, Ag<sub>2</sub>S, etc., in addition to Se) which have a low elec. cond. in the dark and a high elec. cond. when exposed to light. Results of investigations on such substances permit the formulation of the following general characteristics of spectro-elec. sensitivity in solids. (1) The photo-elec. response to an equal energy stimulus is fairly uniform throughout the visible spectrum, terminating in a band or bands of high photo-elec. sensitivity in the extreme red or near infra-red spectrum. (2) The time required to attain equil. of the photo-elec. reaction is selective to wave length and is much shorter for visible radiation than for infra-red rays. (3) The effect of increasing the intensity of the radiation stimulus is to produce a greater increase in the photo-elec. response to the long waves than to the short waves, with a resultant shift of the max. of the spectral photo-elec. sensitivity curve towards the long wave lengths. (4) Decreasing the temp. of the substance greatly increases the intrinsic photo-elec. sensitivity throughout the whole spectrum and shifts the maximum sensitivity towards the short wave lengths.

W. F. MEGGERS

Photoelectric observations on insulating metallic sulfides. B. GUDDEN AND R. POHL. *Z. Physik* 2, 361–72 (1920); cf. *C. A.* 14, 3363.—Homogeneous crystals of ZnS, CdS, and HgS under the influence of light show a strong selective conductivity with large electric fields. The conductivity is established immediately, except when there are chemical changes. The increase of the cond. in the region of long wave lengths coincides with the limit of the optical absorption band.

F. C. HOYT

**Remarks on the distribution of intensity in spectrum series.** J. FRANCK. *Z. Physik* 1, 2-10(1920).—Recent expts. on the distribution of intensity in spectrum series and its dependence upon the pressure of the gas have given results which cannot be brought into accord with Bohr's original explanations, such as that of the absence of the higher members of the Balmer-series in the spectra of discharge tubes and their presence in the spectra of stars. In this theoretical paper the author attempts to lead to an explanation of these phenomena upon the basis of the Bohr atom, noting that the absence or presence of the members of the series depends in the first place upon the arrival of the energy corresponding to the frequency. Their absence in the spectra of ordinary vacuum tubes is to be laid to the too small velocity of the electrons. If a large p. d. is employed to obtain the necessary velocities, the great disturbance of the electronic orbits by the enormous number of ions results in greatly broadened lines. The author proposes that the light emitted by a gas through which a discharge is passing be looked upon as originating in the atom excited by electronic bombardment and not as due to a recombination of opposite ions. He considers also the case of mixtures of two monatomic gases.

C. S. BRAININ

**Series spectra of the elements.** N. BOHR. *Z. Physik* 2, 423-69(1920).—An address, to appear in more detail later. If the stationary states are ellipses in which the energy depends on the major axis only, the emission lines of the Rydberg formula may be thought of as harmonic components of the motion of the electron, at least in the region of large orbits and slow vibration. On this "correspondence principle" if an external field introduces new periodic components into the electron motion it will also introduce new harmonics, which are new spectral lines. In the case of electric and magnetic fields this is shown to lead to the same results for the Stark and Zeeman effect as have been obtained by Epstein, Schwarzschild, and Sommerfeld, who considered the motion as quasi-periodic and quantized each coordinate separately. The method is also useful in treating the spectra of elements of higher atomic number.

F. C. HOVR

**The spectrum of mercury vapor.** J. K. ROBERTSON. *Queen's Univ. Science* 52, 386-7(1920).—A continuous Hg spectrum similar to that observed by C. D. Child (*C. A.* 14, 3367) was obtained on repeating and confirming the expts. of J. Kowalski (*C. A.* 8, 1694). The failure of the light to appear at higher temps. is probably due to the diminished energy of collision resulting from the shortened path caused by the greatly increased vapor pressure at the higher temp.

F. O. A.

**A diamagnetic phenomenon in glowing nitrogen and magnetic behavior of its band-spectrum.** W. STREUBING. *Physik. Z.* 20, 512-21(1919).—A qual. description of the effects observed with pure nitrogen in a magnetic field. For observing the diamagnetic effect the discharge tube passing through the axis of the magnet was expanded in the space between the poles into a bulb which was flattened in a direction at right angles to the field. Without magnetic field, the glow is reddish at low currents and becomes more orange at higher currents. In the above tube and in a magnetic field, a blue glow appears as a central core with the red glow around this, and in the flat bulb a bluish disc is observed at right angles to the magnetic field. S. discusses the probable nature of the molecules exhibiting this diamagnetic effect. Photographs of the spectra observed are also given in the original.

SAUL DUSHMAN

**The series spectrum of helium.** A. LANDÉ. *Physik. Z.* 20, 228-34(1919).—To account for the series spectrum of helium and the alkali metals L. considers the energy relations in the case of a single electron rotating in an  $n$ -quantic orbit with  $(Z-1)$  electrons in an inner ring close to the nuclear charge  $+Ze$ . The outer electron must cause an eccentric shift of the inner ring, and this would affect the energy relations involved according to the quantum theory in the emission of spectral lines. L. considers a helium atom in which the two electrons rotate in concentric (but not neces-

sarily coplanar) rings or elliptic orbits and calculates the frequencies of the spectral lines possible with such an arrangement. The agreement with the observed series is, however, only moderate.

SAUL DUSHMAN

**Disturbance theory of the helium atom.** A. LANDÉ. *Physik. Z.* 21, 114-22(1920).—The same subject (see preceding abstract) is discussed in this paper which is extremely mathematical and difficult to abstract. The disturbing effects of the inner electron on the outer, in the case of elliptical orbits, are considered. This would give rise to the fine structure of the spectral lines. Here again, the agreement is not very satisfactory.

S. DUSHMAN

**The effect of the electric field on the lines of argon and oxygen.** E. BÖTTCHER AND F. TUCZEK. *Ann. Physik* 61, 107-12(1920).—According to Stark's theory the electric force inside an atom due to an external field must be weakened by the presence of loosely bound electrons on the outer ring. Since the inner electrons produce the series spectra, it is to be expected that the effect of an electric field on these spectral lines must decrease with increase in number of electrons in the atom. The authors compare in this respect the effect of an electric field on the series spectra of A and O<sub>2</sub>. The method used was the same as that of Stark, Hardtke and Leibert (*C. A.* 13, 1558). In the case of A no effect was observed. O<sub>2</sub> gives a spark as well as an arc spectrum. According to Stark, the first is due to the presence of O<sup>++</sup>, while the latter is due to O<sup>+</sup>. In the case of the spark spectrum no effect due to the electric field could be observed, while in the arc spectrum a shift towards the red was obtained for lines of the first triplet side series; moreover a series of new lines was observed which possess a marked intensity only in the electric field and are shifted by it towards the violet.

SAUL DUSHMAN

**Absorption of light by gas.** G. RIBAUD. *Ann. phys.* 12, 107-226(1919).—Gases and vapors show continuous absorption for wide spectral regions as well as very fine absorption lines, but quant. data are lacking, especially for bands not obeying Kirchhoff's law. The regions of continuous absorption found in the infra-red have not led to conclusive results. Quant. study of the 2 categories of absorption made on a single substance should furnish information on theories of absorption and permit in particular to decide if the kinetic theory is applicable to wide continuous regions. Simple substances should be chosen and this study has accordingly been made on halogen vapors, principally on Br vapor which had already been observed qualitatively. The first part of the paper contains a succinct statement of the theoretical and exptl. facts now known on the absorption of light. The second part gives detailed results of measurements made on the continuous absorption of Br and Cl vapors and is preceded by a critical study of the methods of measuring absorption in the ultraviolet. The measurements are made by photographic photometry of the spectrum of a Nernst lamp obtained with a quartz spectrograph, quartz cells of Br or Cl of known pressure, temp. and purity being interposed in the optical path. Br possesses an absorption spectrum of very sharp lines from  $\lambda = 0.500\mu$  to the extreme infra-red and a region of continuous absorption between  $0.520\mu$  and  $0.340\mu$  while Cl gives continuous absorption between  $0.450\mu$  and  $0.280\mu$  and sharp lines between  $0.535\mu$  and  $0.480\mu$ . The max. absorption in the case of Br at  $16^\circ$  is found to be at  $0.421\mu$ . The coeff. of absorption is proportional to the pressure and the presence of non-absorbing gases (CO<sub>2</sub>, O or H in excess of 100 atms.) neither widens the absorption band nor changes the wave length of the max. These results disagree with measurements made in the infra-red. The max. of absorption was found to move toward longer waves as the temp. increased; at  $620^\circ$  it was at  $0.433\mu$ . Measurements on Cl verified the law of proportionality of absorption coeff. to pressure from 10 to 76 cm. and fixed the max. absorption at wave length  $0.338\mu$ . The third part is devoted to a general discussion of continuous absorption in extended regions and it is concluded that the kinetic theory of absorption

is not applicable; the mechanism of absorption must be sought for in the interior of the mol. itself. An attempt is made to show that in particular the extinction may be explained by perturbations of the electron in the interior of the mol.; this conception permits an explanation of the fact that the widths of the bands increase from ultra-violet to infra-red. It is shown also that the law of proportionality of the width of the band to the square root of the abs. temp., enunciated by Königsberger must be rejected. In the fourth part a study of the fine absorption lines of Br is undertaken with the following results: The law of proportionality of line width to pressure of a foreign gas is established; quant. measurements of the coeff. of absorption are made showing that if the number of absorbing mols. is negligible compared to the number of inert mols., the coeff. of absorption varies inversely as the pressure in accord with the kinetic theory. Br lines were examd. in a magnetic field but those examd. showed less than 0.01 Å. Zeeman effect in a field of 20,000 gauss. However, there are bright lines in the rotatory magnetic spectrum corresponding to all the lines in the absorption spectrum, and the rotatory phenomenon observed in the neighborhood of absorption lines of the halogens is assumed to be due to a feeble Zeeman effect which is too small to be measured directly.

W. F. MEGGERS

Displacement interferometry by the aid of the achromatic fringes. CARL BARUS. *Publications of the Carnegie Institution of Washington*, No. 249, Part III, 100 pages (1919).

—Displacement interferometry with achromatic fringes is discussed in connection with measurement of long distances, small angles, elastic constants, the Newtonian const., vibrating systems, etc. (discrepancies, anomalies, etc.).

W. F. MEGGERS

Displacement interferometry by the aid of the achromatic fringes. CARL BARUS. *Publications of the Carnegie Institution of Washington*, No. 249, Part IV, 172 pages (1919).

—Description of interferential contact lever and application to observations of elastic deformations, elongation due to magnetization, etc. Other expts. and suggested expts. on the pressure variation of specific heat in liquids, electro-dynamometry of alternating currents, the ether drag, gravitation, etc.

W. F. MEGGERS

The character of light radiations from metallic vapors of magnesium, copper and manganese under the selective action of thermo-chemical and thermo-electrical excitation. G. A. HEMSALECH. *Phil. Mag.* 40, 296-316(1920).—The two modes of excitation, chem. and thermo-elec., have been described in a previous paper (*C. A.* 13, 281). In the present work a single plate carbon resistor, on which the substance under examination is placed, is raised to a high temp. electrically. Tables are given showing the intensities of the various spectral lines, in the fringe (thermo-elec.), and in the luminous vapor (thermo-chem.) as well as those in the cone and in the mantle of the air coal-gas flame and in the capacity spark, for Mg, Cu and Mn. For Mn the arc lines are also given.

S. C. L.

Note on a very convenient method of accurately focusing and adjusting the image of a laboratory light source upon the slit of a spectrograph. G. A. HEMSALECH. *Phil. Mag.* 40, 316-8(1920).—The method consists in illuminating the slit strongly so that its image focused by the objective can be viewed by the use of a hand lens. The spark gap is then shifted until any desired part of it coincides with the image of the slit. The object lens can also be oriented in the same way.

S. C. LIND

Fundamental phenomena in electron tubes having tungsten cathodes (LANGMUIR)

4. Crystal structures of some carbonates of the calcite group (WYCKOFF) 8.

PLANCK, MAX: *Das Wesen des Lichts*. Berlin: Julius Springer. 22 pp. M. 1.60. For review see *Physik. Z.* 21, 528(1920).

SALVADORI, R.: *L'atomismo*. Appendice alla VIII Edizione degli elementi di chimica. Firenze: Le Monnier. 15 pp.

## 4—ELECTROCHEMISTRY

COLIN G. FINK

Dorsey A. Lyon, metallurgist of note. PAUL WOOTON. *Eng. Mining J.* 110, 997(1920); 1 illus.—Biographical sketch. C. G. F.

Ernest S. Jeffries. ANON. *Elec. Rev.* 77, 744(1920); 1 illus.—Brief biographical sketch. C. G. F.

Charles Ernest Acker. ANON. *Elec. World* 76, 1091(1920).—Died Oct. 18, 1920, 52 yrs. old. Inventor of process for  $\text{SnCl}_4$ ,  $\text{CCl}_4$ , etc. C. G. F.

Power for electrochemical purposes. JOHN L. HARPER. *Trans. Am. Electrochem. Soc.* 37, preprint(1920). C. G. F.

Location for a New England electrochemical plant. C. T. MAYNARD. *Trans. Am. Electrochem. Soc.* 37, preprint(1920). C. G. F.

Water powers of the Pacific Coast. F. F. FOWLER. *Trans. Am. Electrochem. Soc.* 37, preprint(1920). C. G. F.

Power for electrochemical industries in Scandinavia. J. W. BECKMAN. *Trans. Am. Electrochem. Soc.* 37, preprint(1920). C. G. F.

The electric furnace as a central-station load. R. D. EVANS. *Elec. J.* 17, 873-80 (1920).—The principal factors affecting the cost of power are the amt. of power delivered, the power-factor of the load, the max. demand and the amt. of unbalanced power. The last consideration is dealt with in detail. Balanced power can be obtained in elec. furnace operation (1) by single or polyphase furnaces having motor-generator sets, (2) single-phase furnaces simultaneously, (3) polyphase furnaces with equal impedance between electrodes, (4) polyphase furnaces with unequal impedances between electrodes with static balancing systems, or (5) single or polyphase furnaces with phase balancers. In the future, more attention will be paid to methods of balancing furnace loads and to detg. the cost of supply in unbalanced loads. W. E. R.

A square deal for the electric furnace. H. G. WEIDENTHAL. *Trans. Am. Electrochem. Soc.* 36, 367-72(1919).—Polemical. C. G. F.

The present position and prospects of electric steel in Great Britain. W. S. GIFFORD. *Electrician* 85, 528-9(1920).—A short review. Cf. C. A. 13, 2808. C. G. F.

Direct production of iron and steel from their ores in the electric furnace. J. ESCARD. *Rev. gén. élec.* 6, 681-8(1919); *Science Abstracts* 23B, 134; cf. C. A. 13, 930. —A detailed review. Malleable Fe is first considered and the work of Stassano, Chaplet, and Arnou. Analyses are given of the minerals used and the Fe obtained, also estimates of cost. The second part of the paper deals with steels and the work of Humbert and Heshey. It is concluded that Mo steel can be obtained directly from the mineral on adding molybdenite and Fe-Si. The most suitable furnace is of the arc type.

LOUIS JORDAN

Electric furnaces as melting mediums. E. F. CONR. *Iron Age* 106, 1059-61 (1920).—Elec. furnace installations in the non-ferrous industries in U. S. on Sept. 1, 1920, had increased by 124, or 40%, in 6 mo., while in the steel industries the increase is 11, or 3%. About 33% of non-ferrous units in the U. S. are credited to foundry operations; and at least 50% of the installations in the steel industry, not including metallurgical or ferro-alloy furnaces. There are about 175 furnaces making steel castings, 5 on malleable iron, 2 on Al-steel, 3 on cast high-speed tools and 2 on iron castings. Data covering 12 types of furnaces for non-ferrous melting and 16 for steel industries are given. W. H. BOYNTON

An improved Greaves-Etchells electric furnace installation. E. T. MOORES. *Chem. Met. Eng.* 23, 825-32(1920).—Improvements recently made in the elec. installation of the Greaves-Etchells furnace include the following features: (1) Independent

regulation of electrodes. (2) The surge resulting from a short circuit on one arc is distributed over all primary supply bases. (3) Short circuits and surges are checked without impairing power factor by introducing extra reactance. A complete description of furnace and arrangement of electrical equipment at the plant of the Holcomb Steel Company, Syracuse, is given. Three transformer windings are used. Primary windings are connected in delta to 3 phases of primary supply, secondary windings are connected in star, two legs being connected to two upper electrodes and the third leg to the hearth. The lining of the hearth is composed of equal parts of magnesite and "syndolag," mixed with pitch and tamped 22 in. thick, and burned out with coke fire for 36 hrs. It is removed after 100 to 115 heats. Side walls are of chrome brick; roof of silica. Records of operation are given. Illustrated.

O. A. HOUGEN

**Electric furnaces and the sulfur problem in cast iron.** GEO. K. ELLIOTT. *Iron Age* 106, 919-20(1920); cf. C. A. 13, 1045.—E. has previously described the production of low-S cast iron by a duplex process—refining a cupola-melted metal under a basic carbide slag in a basic bottom elec. furnace. This process often produces Fe containing S so far below 0.05% that the question is raised as to the advantages accruing from S as low as 0.02%. Sulfides in cast Fe, by freezing last, collect in veins along the joints between crystals of Fe. This is true of so small amts. of S as below 0.05%. When the S content is more than 0.05% it shows a remarkable tendency to segregate in spots. This tendency to segregate constitutes the greatest menace of high S. It can be said that the advantage of very low S is that this non-metallic intercrystalline film is reduced to a minimum. Practically, in the present state of testing and measuring the qualities of cast Fe, it is not possible to show positively the advantage in gray-Fe castings with 0.015 or 0.030% S over those with twice these amts. of S. An indirect but significant advantage of low S is that it gives independence of high Mn which not only saves the cost of Mn but reduces machine-shop finishing costs on account of decreasing the hardness derived from the excess of Mn which is often added in anticipation of high S. Ability to desulfurize so thoroughly permits the use of scrap high in accumulated S.

LOUIS JORDAN

**Heat losses in electric steel furnaces.** FRANK HODSON. *Elec. Rev.* 77, 728-8 (1920).—H. estimates that if the amt. of heat lost in the flame escaping from most furnace roofs were taken into account the heat loss would be nearer 30% of the power used than the 18.7% found by Wolfe and de Wysocki (C. A. 14, 3019). The causes of electrode heat loss are faulty electrode joints or defective electrodes, imperfect contact or badly designed electrode holders, penciling off of the electrode at the point of arc and the combining of O from the air with the C of the electrode and the resultant burning of the electrode. Penciling of electrodes is probably the indirect cause of more heat losses than any other feature. To overcome this there has been developed the Brooke electrode economizer, a type of electrode-cooling ring. This economizer depends for its action on the fact that hot gases under pressure if suddenly allowed to expand quickly lose their high temperature. All other cooling rings simply cool the burning gases issuing from the furnace. In the Brooke economizer these gases never ignite. No flame is seen from the top of the furnace and the electrodes do not pencil off. A diagram of the economizer is given.

LOUIS JORDAN

**Heat content of steel and slag from an electric furnace.** A. M. KUHLMANN and A. D. SPILLMAN. *Trans. Am. Electrochem. Soc.* 38, preprint (1920); *Chem. Met. Eng.* 23, 719(1920).—Calorimetric detns. of the heat content of low-C liquid steel as it flowed from a 6-t. Heroult steel-casting furnace, corrected to 0°, gave 303-325 cal. Liquid slag gave 520.5 cal. at 1250°. Solid slag gave  $S_m(0^\circ - t^\circ) = 0.133 + 0.00011t$ ;  $Q(0^\circ - t^\circ) = 0.133t + 0.00011t^2$ .

CHARLES HECKER

**Von Schlegell repelling arc furnaces.** ANON. *Elec. Rev.* 77, 741(1920).—A fuller account of the furnace described in C. A. 14, 3596.

C. G. F.

**Booth rotating electric furnace.** C. H. BOOTH. *Metal Ind.* 18, 456-9(1920); cf. C. A. 14, 495.—A diagram of elec. connections is shown. All grades of Cu alloys have been successfully melted in this furnace. The character of material varying from sweepings, grindings and low-grade foundry washings up to highest-grade ingot metal and virgin pig. In melting brass turnings, the charge is first ignited to burn off oil before electrodes are inserted. This permits complete removal of oil and grease, and is especially facilitated by rotation. In the melting operation of large charges it is usually best to leave the furnace stationary for a brief period, then advancing the shell slowly, finally rotating completely for 10 to 15 min. before pouring. With small charges rotation may be started at once. Booth furnaces are especially adapted for melting finely divided materials because new metal is continually being exposed to the arc. A very limited time is required for preheating the furnace. At the end of a day's run the furnace is charged for the first heat of the following day. This saves considerable heat and prevents the furnace from cooling rapidly. For instance with the furnace empty it cools down to 250° overnight, whereas when recharged it cools down to only 500° in the same period. Automatic control of electrodes is provided for. The refractory linings can be quickly replaced, requiring a shut down of no more than 24 hrs. These linings are supplied in only three pieces in addition to the two bricks required for the doors, thus minimizing the number of joints. The furnace is tilted on end when lined. Heat insulation is packed in between the lining and the shell, a decided improvement over most brass-furnace linings. Complete rotation of the furnace results in even wear and expansion of the refractories, longer life and lower repair cost. Service tests of furnaces at various plants are given. The av. power consumption in a 500-lb. furnace per t. of metal varies 240 to 370 kw. hr, depending upon the nature of the charge and temp. of pouring. A small 250-lb. furnace is proving of service where a large number of mixes are used and where the daily output is small. O. A. HOUGEN

**Secondary conductors for electric furnaces.** EDWARD T. MOORE. *Elec. J.* 17, 422-7(1920).—A description of the method of reducing lengths of cable in old rocker-type Heroult furnace installations as carried out at the Holcomb Steel Co., is described. The operating performance of this furnace has been improved by reducing the melting period 20 min. and the melting cost by 50 cents per ton. Drawings and photographs are given.

W. E. RUDER

**Some phenomena observed in electric furnace arcs.** J. KELLEHER. *Trans. Am. Electrochem. Soc.* 38, preprint (1920).—The form of arcs is described for a melt of Fe-Ni alloy covered by an acid  $\text{CaSiO}_3$  slag, using a Siemens single-electrode furnace with d. c., when the electrode was negative also when it was positive, and when large currents were allowed to flow. The effect of the structure of the arc on automatic regulation of furnace current is also given.

CHARLES HECKER

**Direct current used for furnace.** CARL REED. *Foundry* 48, 775(1920).—Application of direct current to steel melting has been successful. For good operation it is important to have the charge hot in starting. This is done by permitting the charge to stand overnight in the sealed furnace after a day's run. No greater amts. of ferro-Si and Al are required than in a.c. operation. Normal capacity is 1250 lbs. of steel scrap, with 4 lbs. ferro-Si,  $\frac{1}{2}$  lb. Al and 6 lbs. of ferro-Mn. About 5 lbs. of coke and a handful of CaO are used to prevent oxidation. As many as 6 heats can be run in 14 hrs.

O. A. HOUGEN

**Wild-Barfield electric furnace.** LANCELOT W. WILD. *Chem. Met. Eng.* 23, 699-700(1920); illus.—Wild-Barfield elec. furnaces for the heat treatment of steel are manufd. in three types—radiation, muffle, and salt bath. All types are fitted with the magnetic pyroscopic detector. A refractory lining is wound with a helix of Ni-Cr wire. Outside this is insulation and a case of sheet Al. Outside the Al case is wound a single



layer coil of enamel-insulated Cu wire in series with an indicator. This outer winding is the pyroscopic detector winding. The inner winding acts both as the heat generator and as the primary of a transformer, of which the detector winding forms the secondary. A compensator obviates disturbing effects of fluctuations in the voltage or periodicity of the main line current. When using a. c., as soon as a piece of cold steel is placed in the furnace the voltage of the detector coil is increased and the pointer of the indicator is deflected. When the steel arrives at the beginning of the  $Ac_2$  range of temp. it begins to lose its magnetic susceptibility and it has lost practically all when it has passed completely through this range. At this point the original balance of the detector coil and indicator is regained and the pointer of the indicator returns to zero. If the steel is now withdrawn and quenched the finest grain is obtained without fail. On d. c. circuits a d. c. galvanometer with mirror and scale is used in place of the a. c. indicator. When cold steel is placed in the furnace a momentary throw of the galvanometer is observed, returning at once to the zero. The steel heats and at the beginning of the  $Ac_2$  range a d. c. is again obtained in the detector winding, lasting until the steel has passed entirely through this range. The galvanometer spot then returns to zero. This is the signal for removal of the steel.

LOUIS JORDAN

**Step induction regulator for electric furnaces.** PHILIP B. SHORT. *Trans. Am. Electrochem. Soc.* 37, 35-42(1920) Preprint.—A description of the most recent development in elec. control equipment for elec. furnaces. Cf. C. A. 14, 3196. F. H. H.

**Automatic regulation of electric arc furnaces.** G. Y. ALLEN. *Elec. J.* 17, 397-401(1920).—Following a general description of the various types of regulator, a new type is described in which the motor speed bears a definite relation to the value of the regulated quantity. It uses both the current in each electrode and the voltage of each phase to neutral on the same control elements. It is suitable for any type of furnace because the regulator forces a balance of all arc voltages for balanced electrode currents. Photographs, diagrams and power curves are given.

W. E. RUDER

**Automatic regulation of electric arc furnaces.** ANON. *Elec. Rev.* 77, 243(1920).—Speed of regulation is the governing factor in economical production in elec. furnaces—a new type of regulator is described and illustrated.

W. E. RUDER

**Electric arc-furnace regulator.** ANON. *Elec. Rev.* 77, 624(1920); *Chem. Met. Eng.* 23, 939(1920).—The restoring speed of the electrodes with this regulator is approx. proportional to the current deviation from normal, thus giving rapid operation. Arc-voltage being a factor of the control, each electrode acts independently and gives equal arcs. No under-voltage relay trips are necessary. Starting on a cold charge, it is only necessary to throw on the control, and complete automatic regulation takes place.

F. H. HOTCHKISS

**Physical characteristics of specialized refractories. III. Electrical resistivity at high temperatures.** M. L. HARTMANN, A. P. SULLIVAN AND D. E. ALLEN. *Trans. Am. Electrochem. Soc.* 38, preprint (1920); *Chem. Met. Eng.* 23, 721(1920).—Curves and data are given showing elec. resistivities of 9 commercial refractories at temps. up to 1500°. Results are necessarily only approx.

CHARLES HECKER

**Consumption of electrical energy in the manufacture of electrochemical products.** P. BRUNET. *Rev. gen. elec.* 5, 913-23(1919).—Data are given on the consumption of elec. energy per ton of product and tons produced per kw. yr., as obtained from a variety of sources. Al, Cu, Cl, NaOH, chlorates, electrolytic, metals Fe and steel refining, ferro-alloys, graphite,  $CS_2$ ,  $HNO_3$ ,  $CaC_2$ , carbides and cyanamides are considered in detail as regards efficiency based on theoretical production. In most cases pre-war figures are used.

W. E. RUDER

**Magnesium.** CHARLES VICKERS. *Brass World* 16, 330-3(1920).—V. reviews the patent literature and briefly discusses the various applications of the metal and its alloys.

C. G. F.

**Improvements in the industrial production of oxides of nitrogen in arc furnaces.** F. GROS. *Compt. rend.* 170, 811-3(1920).—G.'s expts. prove that the output of arc furnaces is materially increased by the use of dry air. With moist air, theoretical production is about 120 g. of  $\text{HNO}_3$  per kw.-hr. and the usual actual production is only 65 g. or 54%. With dry air of 50% N and 50% O content the theoretical limit is 264 g. per kw. hr. and G. has obtained up to 90 g. Using a 300-kw. furnace the yield was increased 50%. A closed circuit with sepn. of the  $\text{NO}_2$  by low temp. was used.

W. E. RUDER

**The production of hydrogen and oxygen by electrolysis.** T. A. ROSS. *Elec. Rev. (London)* 86, 4-8(1920); *Science Abstracts* 23B, 241(1920).—A fully illustrated description of a military plant at Rouen, France, where advantage was taken of the slope of the land, the principle being that gas travels upward, while liquids descend. All starting-air receivers were inter-connected and air-piping was duplicated where necessary to minimize possible damage by bomb splinters. Specially large silencers of soft brick were provided for the Diesel engines. Two Diesel engines of 550 h. p. driving two Crompton compound interpole d. c. generators joined to a 480-v. 3-wire system. 52 groups of 50 cells each, of which 4 are in series, are of the Geeraerd type; working without porous diaphragm. Each cell contains two dished cast-Fe plate electrodes with corrugated surface and insulated from each other by pieces of ebonite. Two systems of vanes sloping downwards toward the center of the cell leave free spaces where the diaphragm would be in other types of cells. Each plate is cast with two holes in the upper flange which forms conduits for the gases when cells are bolted together. Gases are purified by passage through wire coils of Pt and platinized fiber, kept at red heat by the current—to a purity of 99.7%, if necessary.

W. H. B.

**Hydrogen overvoltage.** EDGAR NEWBERRY. *J. Am. Chem. Soc.* 42, 2007-11 (1920).—N. criticizes MacInnes, Adler and Contieri (*C. A.* 13, 1792, 2798) in that their expts. and reasonings have serious and invalidating faults: (1) overvoltage was wrongly defined and its measurement confused with p. d. due to transfer resistance; (2) the true Pt overvoltage depends on its state of valence, not the character of its surface, which they sought to explain; (3) no account was taken of the gas film of adhering mols., which is affected by surface conditions but is not related to overvoltage; (4) effects of valence and many similar phenomena readily accounted for by the hydride and higher-oxide theory, are quite unexplained; (5) the observed effects of external p. were related to transfer resistance, not overvoltage. The study of this investigation, properly considered, is interesting and valuable. N. relates his previous work on this subject.

F. H. HOTCHKISS

**Electrolytic zinc.** SAMUEL FIELD. *Chem. Trade J.* 67, 316-7(1920).—To help retain and develop the Brit. Zn industry, F. points out the results of modern research. Tests carried on at Swansea over 4 yrs. culminating in a plant of  $1\frac{1}{2}$  tons daily, indicate that the electrolytic process is (1) entirely practicable for the complex ores encountered, and (2) cheaper and more efficient than the distn. method. The cyclic process utilizes  $\text{H}_2\text{SO}_4$  recovered from calcining the ore, for leaching, together with acid sulfate liquor from electrolytic cells. Neutralization and complete purification of soln. before electrolysis is the most important step. As, Sb, Cu, Cd; Fe, Ni, and Co are removed and recovered when profitable. The soln. of 8% Zn and 3%  $\text{H}_2\text{SO}_4$  is electrolyzed 2 days at 25-30 amp. per sq. ft. using Pb anodes and Al cathodes. When Zn drops to 3% the liquor returns to leaching tanks. Mechanical handling is used practically throughout. The very pure (99.95%) product has opened fields for new uses.

F. H. HOTCHKISS

**Electrolytic iron deposition.** W. A. MACFADYEN. \* *Trans. Faraday Soc.* 15, Pt. 3, 98-133(1920).—To obtain data for building up an industrial process, M. reviews and continues his expts. on Fe deposition (cf. *C. A.* 14, 498). For rapid work 65°

appears to be most suitable, allowing a c. d. of 100 amp. per sq. ft. and 0.01-0.02 N acidity. Usually the max. c. d. giving satisfactory deposits is a direct function of the concn. of the electrolyte. A deposit over  $\frac{1}{8}$  in. thick was obtained. The deposition of hydroxides is chiefly due to presence of org. impurities. Free charcoal removes these, and permits good deposits in neutral solns., i. e., acidity is unnecessary. Several tables give the adherence and character of the deposits as shown by tests of bending, squeezing, impact, sawing, and grinding. Heat treatment was more rapid with finer cryst. deposits, as illustrated by numerous excellent photomicrographs. Voluminous discussion is appended.

F. H. HOTCHKISS

The industrial future of electro-deposited iron. W. E. HUGHES. *Electrician* 85, 530-2(1920); cf. C. A. 14, 2446.—Although worked out by Jacobi as early as 1869, the process of Fe deposition has been neglected in England, in contrast to the activity in other countries (numerous lit. references). Progress during the war was slow. A neutral concd. soln. of  $\text{FeCl}_2$  and  $\text{CaCl}_2$  at 90-110°, and 180 amp. per sq. ft. of cathode has been used commercially for a no. of yrs.; a concd. soln. of Na and Fe sulfates acts similarly but is still exptl. Several lines of research are now under way.

F. H. HOTCHKISS

The reactions of the lead accumulator. D. A. MACINNES, L. ADLER AND D. B. JOUBERT. *Trans. Am. Electrochem. Soc.* 37, 383-90(1920).—Tests were made to det. the no. of  $\text{H}_2\text{SO}_4$  gram-equivalents consumed per faraday passing in the discharge of a Pb accumulator. The first factor was detd. by the change in acid density; the second by the Kelvin potentiometer method. The value found varied from 1.3 to 1.8, the av. being about midway between those required by the "double sulfate" theory of Gladstone and Tribe, and Fery's explanation involving a higher Pb oxide, cf. C. A. 10, 2845. Careful analysis of the material in charged anode showed  $\text{PbO}_2$  only. Furthermore an electrode of chemically prepd.  $\text{PbO}_2$  gave the same potential as an accumulator anode, thus failing to confirm any of Fery's detns. To explain the small amt. of acid reacted upon per faraday of current, the formation of intermediate oxides and basic compds. within the anode pores is suggested, where very dil. acid is present during discharge. Numerous references are appended.

F. H. HOTCHKISS

Storage battery practice in central-station service. J. L. WOODBRIDGE. *Elec. Rev.* 77, 674-8(1920).—The reliability of the storage battery is an important factor in standby service. Recent improvements in design and construction are reviewed. In the original installations 14 mm. spacing between plate centers was used but in response to a demand for max. capacity in a given space this was later reduced to 11 mm. This called for a sp. gr. of the  $\text{H}_2\text{SO}_4$  electrolyte of 1.235 in order to provide a sufficient amt. of acid in the cell to correspond with the active material in the plates. The higher the d. of the electrolyte, however, the greater the tendency to sulfatization of the plates and the shorter is the life of the separators. Accordingly, the standard plate sepn. today is 14.3 mm. between plate centers.

C. G. F.

Care of storage batteries. JEAN LAJUGIE. *Electrician* 85, 112(1920).—Elementary instructions.

C. G. F.

A new form of standard cell. C. J. RODMAN AND THOS. SPOONER. *Trans. Am. Electrochem. Soc.* 38, preprint (1920); *Chem. Met. Eng.* 23, 720(1920).—A portable and very compact standard cell of the Cd type was developed, using a concentric container of Pyrex glass with W leads sealed into the glass. Methods of purifying the ingredients and of filling the cell are described.

CHARLES HECKER

Two common failures of the Clark standard cell. E. C. MCKELVY AND M. P. SHORMAKER. *Bur. Standards, Sci. Paper No.* 390 (1920); *Elec. World* 76, 985 (1920).—The causes and effects of the cracking of Clark cells at the amalgam terminal and the formation of gas in the amalgam limb are discussed. It is shown that the cracking of the cell can best be prevented by the very simple expedient of using a cell blank in

which Pt wire previously subjected to the action of Zn amalgam is employed as the negative terminal; also that the effects of gas formation can be minimized through the employment of the smallest excess crystals required to insure satn. at the highest temp. at which the cell is to be used.

**Plating white metal.** CHAS. H. PROCTOR. *Metal Ind.* 18, 414(1920).—Brass and Ag plating of white metal (antimonial-Pb) for lighting fixtures is described. After buffing, the article is dipped in hot alkali followed by a cyanide dip. A Cu plating is next put on by a 5-min. treatment at 3 v. in a Cu cyanide soln. Next the surface is plated for 1½ hrs. at 1 v. in an acid Cu soln. The article is then scratch-brushed for luster or given a cold acid dip. This gives a surface ready for brassing in a Cu-Zn cyanide soln. at 3-3½ volts, or for silvering in a Ag cyanide electrolyte.

C. H. ELDRIDGE

**Electrolysis of solutions of sodium nitrite using a silver anode.** F. H. JEFFERY. *Trans. Faraday Soc.* 15, 16-20(1920).—Since a compd. derived from  $\text{NaNO}_2$  and  $\text{AgNO}_3$  showing evidence of the existence of the complex anion  $[\text{Ag}(\text{NO})_2]$  has been known for years, the reactions that might take place at the anode during electrolysis of  $\text{NaNO}_2$  using a Ag anode were examd. The cell was formed of a 600-cc. beaker, inside which was a porous pot. The cathode, of smooth Pt, was placed inside the pot while the anode, of Stas Ag, was on the outside. The soln. was agitated by a motor-driven stirrer.  $\text{NaNO}_2$  was free of  $\text{SO}_4$  and Cl but contained some  $\text{NO}_2$ . Solns. of 10-70 g.  $\text{NaNO}_2$  per 100 g.  $\text{H}_2\text{O}$  were electrolyzed. In no case was Ag deposited on the cathode, nor was there any Ag compd. present in the catholyte. The anode area was  $2 \times 47.3 \text{ CM}^2$ ; at 0.07-0.46 amps. Ag went into soln. from the anode; the color of anolyte became bright yellow. No O nor other gas was evolved but a solid deposit formed on the anode which appears to be  $\text{AgNO}_2$ .  $\text{NO}_2$ -ions seem to be without effect on the formation of the deposit on the anode. To isolate the salt of complex anion it was found convenient to use an anolyte concn. of 50 g.  $\text{NaNO}_2$  per 100 g.  $\text{H}_2\text{O}$  and a current of 0.072 amps. Electrolysis lasted 55 hrs. The anolyte was evapd. over  $\text{H}_2\text{SO}_4$  in an exhausted desiccator; small, well formed bright yellow crystals were obtained having the compn.  $\text{NaAg}(\text{NO}_2)_2$ . The effect on the reaction of raising the anode potential was also examd.

D. I. GLEIM

**Analysis of plating solutions.** I, II. J. HAAS, JR. *Metal Ind.* 18, 412-4, 484-6 (1920).—H. points out possible errors of manipulation and shows the value of the gallon assay method of volumetric analysis as applied to Ag, Cu, Zn, brass or bronze, Ni, Au, Pb, black Ni thiocyanate and As solns. The nature of the free acidity in Ni solns. is differentiated between  $\text{H}_2\text{SO}_4$ ,  $\text{H}_3\text{BO}_3$ , and citric acid by means of titrations in the presence of litmus, Congo, or tropaeolin indicators. The tests on black Ni thiocyanate solns. are: Ni, with  $\text{NaCN}$ ; Zn, with  $\text{K}_4\text{Fe}(\text{CN})_6$ , and  $\text{HCNS}$  with  $\text{AgNO}_3$ , and on the cyanide black Ni solns.  $\text{As}_2\text{O}_3$ , Cu, Ni, and Zn. No new principles are involved in the titrations.

W. H. BOYNTON

**Industrial applications of electrolytic conductivity measurements.** EARL A. KRELLER. *Trans. Am. Electrochem. Soc.* 38, preprint (1920); *Chem. Met. Eng.* 23, 721(1920).—The use of cond. measurements, modified by using low-frequency current and galvanometer, is advocated for control of many industrial processes, such as measurement of evaporator concns.

CHARLES HECKER

**Neon lamps for stroboscopic work.** F. W. ASTON. Univ. Cambridge. *Proc. Cambridge Phil. Soc.* 19, 300-6(1919).—For accurate graduation and testing of revolution indicators and similar technical purposes the stroboscopic method is probably the most reliable. The illumination problem seems to have an almost ideal soln. in using Ne lamps. The lamps consist essentially of two relatively large glass bulbs containing the electrodes connected by a long capillary tube. The W filament is about 80 cm. long by 1 mm. diameter and is coiled up inside the space containing the

anode. The electrodes are of Al and fairly large. The filling of these lamps has to be very exact and takes about three hrs., three lamps being filled at once. The gas for filling is previously absorbed in charcoal cooled in liquid air. A quantity is admitted to the exhausted lamp which is then sparked for a time, at a p. of 1 to 3 mm. with a small coil. The impure gas is then pumped off with a Toepler Hg pump, a fresh supply of pure Ne gas admitted and the tube run again. The full charge of 5 to 10 mm. of Ne is now admitted and the lamp sealed off. The life of the lamps appears to consist of two distinct periods, the first, during which chem. active impurities derived from the electrodes and walls of the tube are being slowly and completely eliminated and the second, during which sputtering of the Al cathode takes place and the inactive Ne itself slowly disappears until the p. gets too low for use. During the first period the luminosity steadily improves, remaining almost constant afterwards till near the end of the second period when it rapidly decreases. The best results have been obtained from a batch of lamps filled at about 10 mm. p., some with pure Ne, and some with a mixture of Ne and about 10% He. One of the latter lamps had a working life of 3000 hrs. As to the cause of disappearance of gas, it is generally supposed that the gas remains imbedded or absorbed in the layer of spattered Al on the walls of the tube near the cathode, the idea of true chem. combination not being acceptable without very rigorous proof. Reasons for the superiority of Ne vs. other gases is that the bright orange-red glow of the discharge in Ne is composed almost entirely of lines in the region 5700-6700 Å. u., and is in such striking contrast to sunlight that stroboscopic observations can even be made in broad daylight, the ordinary appearance of the rotating disk having merely a gray background added, looking bluish by contrast. The maximum duration of the working flash is one two-millionth of a sec., so that it can be taken as perfectly instantaneous for the purpose employed. Besides measuring velocity of rotation the Ne lamp may be used with advantage in engineering and other problems. A rapidly rotating mechanism such as an airscrew, if illuminated by a lamp the break of which is operated mechanically at each revolution, will appear at rest, flicker being small at speeds well over 1000 r. p. m. The author claims the technical importance of Ne lamps is rapidly increasing.

C. A. S.

New neon lamp. ANON. *Helios* 30, 287(1920); *Z. Beleuchtungsw.* 13, 109(1920).—A small Ne lamp of the Geisler-tube type giving a yellow-red light ranging between 580 and 740  $\mu$  is described.

C. A. S.

The Cooper-Hewitt lamp. II. Development and application. L. J. BUTTOLPH. *Gen. Elec. Rev.* 23, 858-66(1920).—In tracing the development of the lamp since 1901, it is pointed out that during the years 1902-07 the condensing chamber was standardized, Fe was adopted as the positive electrode material, and an automatic starting device was introduced; between 1907 and 1910 a commercial form of a. c. lamp was developed, and in the latter year a fluorescent reflector was put on the market; during the succeeding period a so-called orthochromatic lamp and a quartz Hg lamp were developed, and the problem of operating the a. c. lamp on a high power factor was solved. The shapes and sizes of the Cooper-Hewitt lamps, the quality of light produced, and the use of the lamp in moving picture studios and in textile and other industries are discussed. The paper is very fully illustrated. Cf. *C. A.*, 14, 3601.

H. JERMAIN CREIGHTON

Fundamental phenomena in electron tubes having tungsten cathodes. IRVING LANGMUIR. *Gen. Elec. Rev.* 23, 589-96(1920).—L. shows that the secondary emission of electrons from the glass surface of a discharge tube becomes prominent when the shape of the tube is such that a large proportion of the electrons bombards the walls instead of passing directly between the electrodes. In tubes of special construction the type of discharge can be altered by accentuating the effect of secondary emission. The voltage-current relations of discharge at low gas p. are characterized by three

types: (a) Discharge in which the current is detd. by the initial velocities of electrons. In this region the current, for negative charge on the anode, is controlled by Maxwell's Distribution Law. Between parallel plane electrodes the curve is exponential and if voltage is plotted against log current, a straight line is obtained. The current increase is somewhat more than 2-fold for each increase by 0.2 v. of the anode voltage. (b) Discharge in which the current is limited by the space charge increases with the  $3/2$  power of the voltage. Log volts plotted against log current is a straight line with the slope  $3/2$ . (c) Discharge in the region where the current is independent of the applied voltage but varies with the temp. according to Richardson's equation. The current increases very rapidly with the temp. If the log current is plotted against  $1/\text{current}$ , a straight line is obtained. L. discusses the combination of the 3 characteristics to give the complete, volt-ampere characteristic of an electron tube, illustrating the various types with practical numerical examples for varying conditions of discharge.

S. C. LIND

Some practical operating features of tungsten filament electron tubes. W. C. WHITTE. *Gen. Elec. Rev.* 23, 840-6(1920); cf. C. A. 14, 2298.—In the practical operation of electron tubes unusual or unlooked for effects frequently occur. The limitations and possibilities of these tubes are analyzed with special reference to the filament, grid, plate, bulb and glass, vacuum conditions and tube circuits. The proper protection of the power supply is also discussed.

W. E. RUDER

Basic theory of electron-tube amplifiers. II. MARIUS LATOUR. *Elec. World* 76, 870-3(1920); cf. C. A. 14, 3199.—The historical development of the 3-electrode tube is discussed. Various arrangements of electron-tube amplifiers are analyzed to demonstrate the conditions necessary for max. amplification. In increasing amplification by the use of several tubes arranged in cascade, it is pointed out that not only is watt-power amplified, but also magnetizing or reactive power. H. J. CREIGHTON

An audion frequency vacuum-tube generator. J. A. EYSTER. *J. Am. Inst. Elec. Eng.* 39, 889(1920).—The use of the 3-element vacuum tube for supplying a. c. to a conductivity bridge is described and illustrated. In using a Wheatstone bridge for measuring the conductivity of solns. the induction alternator, the elec. tuning fork, Vreeland oscillator, and the microphone hummer do not give a pure sine wave nor anything approaching it; measurement of solns. of say 500 ohms per cell resistance, cannot be relied upon for accurate readings. The vacuum tube oscillator is the only source of a. c. giving a perfect sinusoidal current; it is const. and reliable; it is capable of a very wide range of pitch adjustment. The Western Electric "V. T. 21" was used; for many purposes the tube "V. T. 1" was found to be satisfactory although of less power (max. plate potential 350 v. vs. 500 v. for V. T. 21). For V. T. 1 a plate voltage of 75 v. was found to be sufficient when measuring the conductivity of electrolytes. Diagrams of connections are shown.

C. G. F.

Corona discharge. E. H. WARNER AND J. KUNZ. *Univ. Illinois Eng. Expt. Sta., Bull.* No. 114, 128 pp.(1919); *Science Abstracts* 23B, 337-9(1920).—A discussion of the results of an investigation to develop a satisfactory theory for corona phenomena. Continuous voltage was obtained by a battery of 50 500-v. 250-watt d. c. shunt-wound generators connected in series, also with H as dielectric. The discharge tube was a cylinder with wire strung along its axis. The visible glow-voltage point may be at or above the critical voltage. When an a. c. voltage was impressed across a tube filled with H, the discharge was similar to that when the wire was negative; which indicated a practically perfect rectification. If the a. c. voltage was increased sufficiently a + discharge formed and then a + arc, proving that the arcing occurred during a half-cycle in which the wire is +. In air of known humidity, the corona was a function of the radius of the wire and atm. p. The time element was important with H and critical v. detns. must be made when normal conditions have been resumed after breaking the

corona circuit. Confinement of air in a tube had a great effect upon the corona current and critical and visible glow-voltage. Characteristic curves of corona in H showed a marked difference between the critical voltage and the voltage at which the corona ceased and differences between points taken with increasing and decreasing current. Moisture raises the starting-point of + corona in air and reduces the — corona. Dry air in a tube reduces the amplitude of vibration for wire + and stops the vibration for wire —. Difference in temp. made a greater difference in current for wire —, both currents showing increase for high temp. The appearance of + and — corona, the effect of the metal constitution of wire and the nature of the surface are discussed and exptl. data given to show that O<sub>3</sub> formation in the corona is related to the forces holding atoms together in molecules. Evidence is also given to show the relation of corona to the arc discharge and an attempt made to explain the pulsating current obtained. The current on abraded and corroded surfaces is larger than for polished surfaces. For low p. the beads have about the same degree of stability for all surfaces, but at high p. beads are more stable on abraded surfaces. A. c. may be perfectly rectified up to 42000 v. by the corona in H. For given gas p. the max. v. that may be rectified is nearly proportional to the radius of the outer cylinder when the radius of the inner cylinder is small by comparison. The increase in gas p. immediately following the appearance of the corona is considered due to ionization rather than to Joule heat. Ionization p. in + corona and any chem. action due to corona are proportional to the corona current. The spark gap in series with the discharge tube affects + and — corona in characteristic striking ways, due to the intermittent current. A Braun tube with hot-CaO cathode was developed for use in observing a weak pulsating current which passes through the arc and the corona tube. The relation of corona to the arc discharge is shown and an attempt made to explain pulsating current. Research is in progress concerning mechanical vibrations of wires and the nature of the elec. wind.

W. H. BOYNTON

**New electrical precipitation treater.** M. SHIBUSAWA AND J. NIWA. *J. Am. Inst. Elec. Eng.* 39, 890-908 (1920).—The new pptn. treater consists of a metal cylinder as the passive electrode and a metal wire suspended along the center of the cylinder as the active electrode; the only difference between this and other treaters being that the electrodes are entirely sep'd. by a dielectric, such as glass, porcelain, etc. The use of glass-covered electrodes prevents the deposition of particles on the electrodes, the corrosion of the active electrode, and sparking between the electrodes. Expts. carried out at the electrochem. lab. at Tokyo and at the Cu refining works at Nikko are described, and the field of practical application of the new treater is discussed. The potential gradient in the treater has been statically solved. The variation of the resistivity of the glass with temp. and with potential gradient, and the free charges on the glass surface have been studied. As the reason why so large a current is permitted to flow through the glass and thus make elec. pptn. possible with glass-covered electrodes, it is concluded that: (1) Resistivity of glass decreases with the temp. as well as with the potential gradient applied; (2) free charges appear on the surface of the glass placed in the elec. field, and these produce in the dielectric a much higher potential gradient than that calcd. by statical considerations; (3) the foregoing causes permit sufficient current to pass through the dielectric to effect pptn. H. J. CREIGHTON

**The new liquid rheostats.** D. C. WAST. *Elec. J.* 17, 483-4 (1920).—The liquid rheostat has very high thermal capacity, ability to dissipate large amts. of heat quickly, and characteristic smoothness of resistance variation. Two electrode assemblies of 3 electrodes each, side by side in a rectangular tank, are provided with a set of weirs for raising and lowering the height of the electrolyte around the electrode. A by-pass shunts a portion of the electrolyte to the cooling tower and back to the electrolyte tank. An adjacent tank carries fresh water. The complete outfit is 6 ft. high with a 4' × 5'

base. Electrodes, connected to slip rings of the main driving motors, have their lower ends resting in grooves in soapstone blocks, thereby holding them in place and insulating them from the bottom of the tank. The electrodes and ground plates are sheet steel of light, rigid construction. At the minimum level, the path of the current is directly through the electrolyte, the lengths and equivalent cross-sections of all these paths are equal and a balanced 3-phase delta-resistance is obtained. The high resistance necessary to limit the current at the start is obtained by wide spacing of electrodes, by narrowing in of the electrodes at the bottom, and by the use of soapstone barriers. The very low max. c. d. and the fact that no circuits are completely broken between electrodes and electrolytes eliminates possible arcing and burning of electrode dips. Sudden resistance changes and concn. of current in the electrolyte off the center of the electrode are prevented by cutting ground plates away in the center. The height of the electrolyte is controlled by 15 long narrow segmental weirs extending lengthwise across the tank, along the edge of the electrodes and resting on one another. Each set of weirs is controlled individually. The electrolyte that is by-passed goes through the cooling tower, where large quantities of heat are rapidly dissipated without damage to the app. The rheostat is designed to allow a third of the normal weight of electrolyte to evap. without lowering the resistance beyond the allowable limit. Normal operation is at const. density with 35% reserve for operation at variable densities.

W. H. BOYNTON

Industrial readjustments of certain mineral industries affected by the war (RIDDELL, *et al.*) 13. Electric insulation materials (ANON.) 13. Construction of thermocouples by electrodeposition (WILSON, EPPS) 1.

COGGESHALL, KENNETH M.: *Modern Electroplater*. New York: Norman W. Henley Publishing Co. 276 pp. \$3.50. For review see *Can. Chem. J.* 4, 318(1920).

FORSTER, A.: *Vom Kienspan bis zur Quecksilberlampe und dem Teslalicht*. Bern: R. Dech & Co. 64 pp. M. 2. For review see *Z. physik. chem. Unterricht* 33, 196(1920).

Electrolyte for primary batteries. G. S. ENGLE. U. S. 1,356,436, Oct. 19. A gelatinous electrolyte is formed of a soln. of NaOH of about 28° Bé. mixed with starch and heated to about 80°. Less starch is used than that which would be necessary to gelatinize an equal amt. of H<sub>2</sub>O.

Storage batteries. P. D. IVEY and A. J. SALISBURY. U. S. 1,354,255-6, Sept. 28. Structural features.

Storage battery electrode. C. AMBRUSTER. U. S. 1,354,086, Sept. 28. Structural features.

Electrode for alkaline storage batteries. R. SAVILLE. U. S. 1,354,513, Oct. 5. Structural features.

Electrode for photoelectric cells. R. IMORI, T. TAKHBE AND THE SCIENTIFIC RESEARCH LAB. Jap. 35,662, Jan. 28, 1920. The electrode consists of a plate of Ag, Pb, Cu or Hg (or a plate of some other metal coated with one of the above metals). The electrolyte consists of an aq. soln. of some halide salt, *e. g.*, KI, KCl, FeCl<sub>3</sub>, CuCl<sub>2</sub>, or HgCl<sub>2</sub>. By exposing the cell to the action of light rays, light energy, it is claimed, is converted into electrical energy without appreciably consuming the electrode metal.

Copper oxides in battery electrodes. G. S. ENGLE. U. S. 1,356,435, Oct. 19. CuO and Cu<sub>2</sub>O are used together in forming electrodes for primary batteries.

Copper oxide electric battery element. G. S. ENGLE. U. S. 1,356,197, Oct. 19. battery element is prepd. by the following method. A mass of flakes of black oxide Cu containing particles of metallic Cu is mixed with a binder such as sugar and



heated sufficiently to reduce the binder to a small residuum. The mass is then subjected to a much higher temp. (preferably about 600°) first entirely to eliminate the binder residuum and then to change the metallic Cu to  $\text{Cu}_2\text{O}$ . The mass is compressed while heated to redness. Battery elements thus prepd. are stated to have unusual durability.

**Dry-cell electric battery.** C. ELLIS and A. A. WELLS. U. S. 1,355,699, Oct. 19. The portion of the depolarizing mass adjacent the Zn container is formed of relatively porous material. During the operation of the battery the formation of an insol. Zn compd. does not retard the operation for a long period.

**Electrolytic production of metals from molten electrolytes.** H. C. M. INGEBERG. Ger. 319,530, Oct. 27, 1917. In a refractory vessel of Fe, coal or other material not affected by the melt, a second vessel is placed which serves as diaphragm and may be made of soda glass, with or without reinforcement. The cathode is mounted in this vessel, while the anode consists of a hollow C cylinder enclosing the diaphragm vessel. Both the catholyte and anolyte consist of a mixt. of NaCl and KCl, which m. about 660°, while the electrolysis is effected at about 700° and with 7-10 v. The diaphragm material acts as an intermediate electrolyte, in that the Na ions carry the current. If Mg is to be isolated, the melt in the anode chamber consists of an equimol. mixt. of NaCl and KCl, while the melt in the cathode chamber consists of a mol. mixt. of  $\text{MgCl}_2$ , NaCl and KCl with about 10%  $\text{Na}_2\text{F}_2$ . The electrolysis is carried on at 700-750° and with 7-10 v.

**Electrolytic production of hypochlorites.** P. PESTALOZZA. Brit. 148,095, Dec. 19, 1919. A vat for the direct production of hypochlorites is divided by transverse partitions having staggered openings in their upper parts for passage of the chloride soln., each partition carrying a bipolar element comprizing an anode plate in one compartment and two vertical cathode plates having their upper edges at different levels and arranged on opposite sides of the anode in the next compartment. Beyond the higher cathode plate which is situated adjacent the inlet opening in each compartment, is immersed a loop from one of a pair of cooling pipes carried along the vats. Circulation of the liquid between the electrodes is thus promoted. The cell with its integral partitions is preferably molded under high pressure from a mixt. of cement, asbestos, and  $\text{H}_2\text{O}$ , and, after drying, is impregnated, while hot, with heavy oils or paraffin. A bath of paraffin and neutral oil at 200° is suitable. Each partition has a central slot in which slides a block of similar material carrying the bipolar electrode element, or the single elements at the ends of the cell. The anodes may be of platinum, and the cathodes of C or graphite. The cooling pipes may be of glass, with rubber connectors at intervals.

**Electrolytic production of copper sulfate.** R. E. BRA. Brit. 147,689, July 8, 1920. Cryst.  $\text{CuSO}_4$  is obtained from commercial waste Cu, which may be placed in a perforated container of wood, earthen-ware, or sheet Pb, suspended in a vat containing dil.  $\text{H}_2\text{SO}_4$ . Lead rods pass into the waste to connect it as anode. Also suspended in the vat are porous pots of asbestos porcelain, earthenware, or other material resisting the passage of Cu ions. Lead cathodes are placed in the porous pots. All the anode containers and cathode pots of a number of vats may be carried by a single framework. During the process the temp. rises, say to 35-40°, and strong  $\text{CuSO}_4$  soln. descends to the bottom of the vats. The salt may be crystd. out by external cooling of the bottom of the vats, and may be collected by trays of Cu, brass, Pb, etc., fitting the vats. Alternatively, metal receptacles with double walls for cooling liquid may be placed in the vats, or the soln. may be discharged continuously or at intervals by siphons and cooled separately. A little finely divided Cu collects in the porous pots and is oxidized, with or without heating, and used to neutralize any free acid in the  $\text{CuSO}_4$ .

**Electrolysis of sodium chloride solutions.** A. LUIS and E. STEINBUCH. U. S. 1,354,428, Oct. 5. In electrolysis of NaCl to form NaOH and Cl or in the electrolysis of other liquids to produce a solute and evolve a gas, the solute is expelled from the electrolytic cell by the buoyancy of the evolved gas. An app. is described.

**Alkaline melts for chemical reactions.** E. BEROVS. U. S. 1,354,451, Sept. 28. A melt containing Na and K or similar melts adapted for use in the manuf. of *indigo* or other *dyes* may be obtained by electrolyzing a melt containing NaOH and KOH with an anode current density of 20–50 amperes per sq. cm. and a cathode current density of below 1 ampere per sq. cm.

**Alkaline gelatinous electrolyte.** G. S. ENGLE. U. S. 1,356,434, Oct. 19. In making a battery electrolyte containing about 10% of starch together with an aq. soln. of NaOH or KOH, the mixt. is thoroughly stirred and heated to a temp. below its b. p. in order to avoid formation of substances which would harden the electrolyte and then is allowed to cool before pouring.

**Electrolytic cell for decomposing sodium chloride solutions.** H. I. ALLEN. U. S. 1,355,116, Oct. 12. The cell produces NaOH, Cl and H.

**Electrolytic apparatus adapted for deposition of copper from crude solutions.** W. E. GREENAWALT. U. S. 1,353,995, Sept. 28.

## 5—PHOTOGRAPHY

LOUIS DERR

**Theory of the oil-pigment process.** WURM-REITHMAYER. *Phot. Rundschau* 57, 29–35(1920).—It is argued that the varying retention of greasy inks by a dichromated gelatin plate exposed under a negative is not due to differences of absorbing power conferred by the varying moisture content of the lights and shadows, but to a difference of surface condition, probably physical, caused by the unequal swelling of the gelatin in the differently insolated areas and the consequent differences in the mechanical strains produced thereby.

L. DERR

**Use of coloring matters of flowers in color photography.** P. R. KÖGEL. *Phot. Korr.* 57, 86–91(1920).—Various benzopyryllium compds. occurring in flowers, themselves stable, can be given high light-sensitiveness, especially by *o*-anethole. As this substance, with other similar ones, may occur as a disintegration-product in plants, these coloring matters (anthocyanins) may thus supply their own sensitizers. Their low covering power, however, prevents their use in the bleach-out process.

L. DERR

**Theoretical basis of a new direct photographic color process with colored substances.** P. R. KÖGEL. *Phot. Korr.* 56, 332–7(1919).—Since only absorbed light is chemically active, a colored enolic compd. which is bleached by light into a colorless keto compd. is affected only by light of a color complementary to its own. The dark-brown sensitive film consists of a mixt. of 3 enolic substances, red, yellow, and blue; in white light it bleaches completely, but retains the corresponding complementary color when acted upon by colored light. Sensitizers may be applied to the mixed colors, and the pictures may be fixed.

L. DERR

**Theory of organic photographic developers.** B. HOMOLKA. *Phot. Korr.* 56, 387–91(1919). When a COOH group is introduced into pyrogallol in the formation of gallic acid, the loss of developing power is explained by the formation of an anhydride by the COOH group and the OH group in *p*-position to it. Pyrogallolcarboxylic acid and catechol-*o*-carboxylic acid, in which such anhydride formation is impossible, are developers.

L. DERR

**State of our knowledge of organic photographic developers.** A. SEYEWITZ. *Bull. soc. franç. phot.* 62, 129–39(1920); cf. C. A. 14, 1267.—A review. Many structural formulas are given.

L. DERR

**Intensifying and reducing pyro-developed negatives by redeveloping.** R. B. WILSON. Eastman Kodak Co. *Bull. soc. franç. phot.* 62, 139-43(1920).—The pyro-developed Ag image is always more or less colored by reduction-products of the developer; and while this brown image is left intact when the Ag is changed to AgBr by a mixt. of  $K_4Fe(CN)_6$  and KBr it disappears when the Ag is changed to AgCl in a chlorinating bath having a permanganate base. In the first case, repetitions of the bromiding and development in alternation produce a progressive intensification; in the second case, a reduction is obtained by chlorinating and redeveloping with a non-staining developer like metol-hydroquinone. The intensification compares favorably with the results of other known methods.

L. DERR

**New method for the measurement of photographic filter factors.** R. DAVIS. *J. Opt. Soc. Am.* 4, 300-4(1920).—Light from one side of a standard lamp in a long box is reflected by two inclined mirrors at the end of the box, falling upon a totally reflecting prism and thence upon a photographic plate. Similar mirrors at the other end and a similar prism send a second beam to the plate, alongside the first, and in some one position of the lamp the intensities of the two beams are the same, if the paths are of equal length and similar optical conditions exist in mirrors and prisms. If now a color filter is placed in one beam, the lamp will have to be moved toward it to obtain equality of effect upon the plate, and the law of inverse squares will give the multiplying factor of the filter on the plate used. In practice the lamp is moved by successive arbitrary intervals, a number of exposures made on the same plate, and the point of equal density obtained by interpolation.

L. DERR

**Color of the image in print-out papers and factors affecting it.** F. FORMSTECHE. *Deut. opt. Wochschr.* 1920, 33-4.—The color of the printing image depends on the size of the pptd. Ag particles, which is affected by the initial condition of the sensitive Ag salt and also by the strength of the printing light and by moisture. Emulsions with a low proportion of citrate give blue images, the particles becoming finer grained and the color redder with increasing quantities of citrate. The redder images have also a softer gradation.

J. S. C. I.

**Renovation of printing-out papers.** A. SPELUZI. *Bull. soc. franç. phot.* 62, 158 (1920).—Papers which have been damaged by too long keeping may be bleached in a mixt. of  $Ca(ClO)_2$  and  $H_3BO_3$ , and dried in the dark. The sensitiveness is restored by a bath of 0.05%  $AgNO_3$ , and when dry the paper is as good as new.

L. DERR

**Relation between photographic density, light intensity and exposure time.** F. E. ROSS. *J. Opt. Soc. Am.* 4, 255-73(1920).—The various formulas expressing the relations between the three factors are studied analytically, and suggestions are given for more precise study.

L. DERR

**Vanishing pictures.** H. LOFFO-CRAMER. *Phot. Rundschau* 57, 265-7(1920).—The image on a daguerreotype Ag film completely converted into AgI, and developed with Hg vapor but not fixed, disappeared completely after several weeks in the dark. The well-known permanence of early daguerreotypes is attributed to the Au toning and the existence under the AgI of a Ag film which holds the Hg in amalgamation.

L. DERR

**Hypo eliminators.** ANON. *Phot. Rundschau* 57, 278(1920).—Dil.  $KMnO_4$ , either acid, neutral, or alk., attacks the Ag image; neutralized  $(NH_4)_2S_2O_8$  or percarbonates may be used.

L. DERR

**Coloring matters for increasing activities of the dry plate for light.** SEIJI NAKAMURA. *J. Orient. Sci. Arts.* 37, 401-7(1920).—A review.

K. K.

LIESSEGANG, R. ED.: *Photographische Chemie in allgemein verständlicher Darstellung*. 4th Ed. newly edited by Karl Kiefer. Leipzig: Ed. Liesegangs Verlag, M. Eger. M. 7, bound M. 9.50. For review see *Phot. Rundschau* 57, 296(1920).

**Colored photographs.** A. TRAUBE. Brit. 147,005, July 6, 1920. For making colored photographs, silver prints toned by inorg. toning methods as with Cu, U, Fe, etc., to produce double salts, can be dyed with org. basic dyestuffs. Prints toned with  $\text{Cu}_2\text{Fe}(\text{CN})_6$  can be dissolved away by a weak soda fixing soln. Methylene blue (1 in 10,000) preferably with the addition of HOAc may be used. Without removing the Ag, very transparent pictures can be obtained by coating with a varnish such as a soln. of damar in benzene.

**Colored photographs.** A. TRAUBE. Brit. 147,103, July 7, 1920. Addition to 147,005 (above). For the process of dyeing Cu-toned images described in the principal patent, dyestuffs of a basic nature which contain a ring such as thiobenzoyl, thiazine, pyronine, oxazine, and acridine dyestuffs, are used to dye the toned image. To correct a too strongly dyed picture, an acid bath may be used, and for intensifying the image a further treatment with a dye soln. is given. It is best to intensify after drying. For the Cu toning bath less than half the usual amt. of Cu and  $\text{K}_3\text{Fe}(\text{CN})_6$  may be used while the normal proportion of K citrate is retained, and the transparency of the picture can be still further increased by using more  $\text{K}_3\text{Fe}(\text{CN})_6$  than Cu.

**Reducing light-sensitiveness of photographic silver compounds.** N. SULZBERGER. U. S. 1,356,236, Oct. 19. Light-sensitive Ag compds. used in photography, such as residual AgCl or AgBr in developed photographic paper or films, are treated with a soln. of  $\text{K}_3\text{Fe}(\text{CN})_6$  in order to reduce or destroy their sensitiveness to light and obtain a permanent image.

**Photographic plate.** F. F. LENWICK, O. BLOCK and ILFORD CO. Japan 35,359, Nov. 19, 1919. Sensibility of photographic plates for color is increased by immersing them in a soln. of 1 g. auramine or coloring matter of iminodiphenylmethane series in 50 l.  $\text{H}_2\text{O}$ .

**Emollient for motion-picture films.** P. WERNER. U. S. 1,354,009, Sept. 28. A mixt. for treating motion-picture films for preventing brittleness is formed of bees-wax 0.5, paraffin 6.5, camphor 1, stearic acid 0.5 and tallow 7.5 parts.

## 6—INORGANIC CHEMISTRY

H. I. SCHLESINGER

**Reaction between selenium monochloride and ethylene.** F. H. HEATH AND W. L. SEMON. *J. Ind. Eng. Chem.* 12, 1109-1 (1920).— $\text{Se}_2\text{Cl}_2$  was prepd. by passing a fairly rapid current of HCl into a soln. of 100 g. Se in 300 g. of 30% oleum gently heated. Yield 130 g. in 2 hrs. The crude product was purified by shaking with small portions of fuming  $\text{H}_2\text{SO}_4$  until the green color of  $\text{SeSO}_2$  no longer appeared in the acid layer. On leading a slow stream of  $\text{C}_2\text{H}_4$  into  $\text{Se}_2\text{Cl}_2$  Se was pptd. and HCl evolved. When the reaction was complete hot  $\text{CHCl}_3$  was added, Se filtered off, and the white needles pptd. on cooling recrystd. from  $\text{CHCl}_3$ . M. p., 118°. Analysis indicated  $\text{C}_2\text{H}_4\text{Cl}_2\text{Se}$ . By analogy with similar reactions of the S compds. the product is concluded to be *symmetrical tetrachlorodiethyl selenide*. This compound, or some by-product, caused water blisters and a sense of drowsiness. A. R. M.

**Sodium phosphide—metallic hydrides.** E. TOMKINSON. *Chem. News* 121, 177 (1920); cf. C. A. 14, 3201.—References to the literature omitted in the previous article are given. A. R. M.

**Formation of addition compounds between 100% sulfuric acid and the neutral sulfates of the alkali metals.** JAMES KENDALL and MARY L. LONDON. *J. Am. Chem. Soc.* 42, 2131-42 (1920); cf. C. A. 11, 3151.—A complete investigation by the f. p. method was made in systems containing anhydrous acid and anhydrous neutral sulfates of K, NH<sub>4</sub>, Na and Li from the eutectic pt. of each system to about 300°. Beside


comps. of undetd. compn. the following were isolated:  $K_2SO_4 \cdot 3H_2SO_4$ , m.  $91.5^\circ$ ;  $K_2SO_4 \cdot H_2SO_4$ , m.  $218.6^\circ$ , exists in 3 modifications with transition pts. at  $182^\circ$  and  $202^\circ$ ;  $(NH_4)_2SO_4 \cdot 3H_2SO_4$ , m.  $48.0^\circ$ ;  $(NH_4)_2SO_4 \cdot H_2SO_4$ , m.  $146.9^\circ$ ;  $2Na_2SO_4 \cdot 9H_2SO_4$ , unstable at m. p.  $80^\circ$  (extrapolated) transitions at  $57^\circ$  and  $58^\circ$  to 2 modifications of  $Na_2SO_4 \cdot 2H_2SO_4$ , both unstable at m. p., with transitions at  $95^\circ$  and  $109^\circ$  to  $Na_2SO_4 \cdot H_2SO_4$ , m.  $186^\circ$ ;  $Li_2SO_4 \cdot 7H_2SO_4$ , m.  $13.6^\circ$ ;  $Li_2SO_4 \cdot 2H_2SO_4$ , unstable at m. p., transition at  $50^\circ$  to  $Li_2SO_4 \cdot H_2SO_4$ , unstable at m. p.  $170.5^\circ$  (extrapolated). The comps.  $K_2SO_4 \cdot 6H_2SO_4$  and  $Na_2SO_4 \cdot 8H_2SO_4$  claimed by Bergius (C. A. 4, 1704) could not be confirmed.

A. R. M.

Reaction between chlorine and ammonia. II. W. A. NOYES AND A. B. HAW. *J. Am. Chem. Soc.* 42, 2167-73 (1920); cf. *J. Am. Chem. Soc.* 23, 460 (1901); C. A. 7, 2522.—The anhydrous gases combine according to  $4NH_3 + 3Cl_2 = 3NH_4Cl + NCl_3$ . A considerable part of the  $NCl_3$  decomps. into  $Cl_2$  and  $N_2$  either directly or by reaction with  $NH_3$ . The ratio mols.  $Cl_2$ :mols.  $NH_3$  may be varied within wide limits without affecting, appreciably, the ratio mols.  $NH_3$ : mols.  $NH_4Cl = 4:3$ .  $Cl_2$  reacts with  $NH_4Cl$  little or not at all. III. Probable formation of trichloro-ammonium chloride. W. A. NOYES. *Ibid* 2173-9.—Anhydrous  $HCl$  converts  $NCl_3$  quant. to  $NH_4Cl$  either in soln. in  $CCl_4$  or without solvent, demonstrating that the decompn. is not an hydrolysis, in absence of water, and probably not an hydrolysis in presence of water. Evidence is presented that the action consists in formation of  $NCl_3 \cdot HCl$  which loses an atom of  $Cl^+$  combined with an atom of  $Cl^-$  and that formation of  $NCl_3$  by action of  $Cl_2$  on an  $NH_4$  salt (sulfate or phosphate is preferable to chloride) is the reverse action. In the latter  $NH_4Cl$  or  $NHCl_2$  is formed as well as  $NCl_3$ .  $NCl_3$ , free from other chloroamines, is prepd. better by treatment of an  $NH_4$  salt with  $HClO$  than with  $Cl_2$ .  $NCl_3$  can be sepd. from a soln. in which it is prepd. by passing a current of air through the soln., drying air and vapor of  $NCl_3$  with concd.  $H_2SO_4$  and condensing  $NCl_3$  by a freezing mixt.

A. R. MIDDLETON

Constitution of the silicates. JOHANN JAKOB. *Helvetica Chim. Acta* 3, 669-704 (1920); cf. C. A. 14, 917.—By a comprehensive comparative study of the transformation products of minerals their constitution is deduced. The silicates are here formulated according to the Werner principles. The prototype is considered to be  $[SiO_6]R_4$  which dissociates easily at high temps. to coordinatively unsatd. penta- and tetraoxysilicates which easily polymerize to higher forms, e. g., asperolith,  $CuH_6SiO_4$ ; chrysocolla,  $CuH_6SiO_4$ ; diopside,  $CuH_6SiO_4$ . Adjacent pairs of O atoms at the octahedron corners can add  $SiO_2$  forming mono-, di- and tri-hexoxysilicates and pairs of H atoms in the outer sphere have residual affinity sufficient to hold water mols.,

forming the group ; e. g., serpentine,  $[SiO_5 \cdot SiO_6]^{Mgs.}_{H_2O, OH_2}$ ;

$[SiO_5]^{Mgs.}_{SiO_6}$  diopside,  $[Si \begin{pmatrix} O & O \\ & Si \\ O & O \end{pmatrix}]^{Mgs.(Fe)}_{Ca_3}$ . The octahedral formulation

predicts the possibility of optical isomers in the two latter cases. Further  $SiO_2$  groups can add upon 3  $SiO_2$  groups of  $[(SiO_2)_3 \cdot SiO_6]R_3$  forming the types  $[(O_3SiO_2)_2Si(O_3SiO_2 \cdot SiO_6)]R_3$ ;  $[(O_3SiO_2)Si(O_3SiO_2 \cdot SiO_6)_2]R_3$  and  $[Si(O_3SiO_2 \cdot SiO_6)_3]R_3$ . Al,  $Fe^{III}$ ,  $Fe^{II}$  and Mg can act as central atoms of the octahedron and add  $SiO_2$  octahedrons by common adjacent pairs of O atoms forming silicato-salts; e. g., muscovite,  $[Al(SiO_3)_3]Al_2KH_2$ . Amphiboles are considered polymerized pyroxenes which are unstable except in water-rich magmas and by loss of secondary  $SiO_2$  groups form olivine:  $3[(SiO_2)_2 \cdot SiO_6]^{Mgs.}_{pyroxene} \rightleftharpoons$

$[Mg((SiO_2)_2 \cdot SiO_6)_3]^{Mgs.}_{amphibole} \rightleftharpoons [Mg(SiO_6 \cdot SiO_2)_3]^{Mgs.}_{olivine} + 6SiO_2$ . Tourmaline is formu-

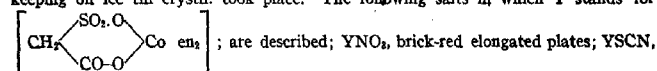
lated as  $4[\text{Al}(\text{SiO}_2)_2]\text{R}_3 + 1 \left[ \text{Al} \begin{pmatrix} \text{OBO} \\ \text{O} \\ \text{OBO} \end{pmatrix} \right]_2 \text{Al}$ . Hydrolysis processes are discussed at length.

A. R. MIDDLETON

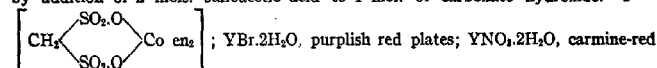
**Nature of the  $\beta$ -ferricyanides and the  $\beta$ -ferrocyanides.** S. H. C. BRIGGS. *J. Chem. Soc.* 117, 1026-34(1920); cf. Belluci, *C. A.* 14, 3028.—Analysis of Locke and Edwards' salt (*Am. Chem. J.* 21, 193, 413(1899)) pointed to the formula  $3\text{K}_3\text{Fe}(\text{CN})_6 \cdot \text{K}_3[\text{Fe}(\text{CN})_5\text{H}_2\text{O}]$ ; the compd. gave Hofmann's reaction (*Ann.* 312, 1(1900)) for  $\text{Fe}(\text{CN})_6$ , derivs. and a soln. of Hofmann's  $\text{Na}_3[\text{Fe}(\text{CN})_5\text{H}_2\text{O}]$  with 3.5 mol. equivs. of  $\text{Na}_3\text{Fe}(\text{CN})_6$  was pptd. at once by  $\text{Bi}(\text{NO}_3)_3$ , proving free ferricyanide present, while a soln. with 2.5 mol. equivs. gave no ppt. after a week and hence contained no free ferricyanide. The deep red crystals obtained by oxidation of ferrocyanide by  $\text{Cl}_2$  are, therefore, mixts. of ferricyanide and the double salt of ferricyanide and aquopentacyanoferrate (Cambi, *C. A.* 5, 2228). Similarly  $\beta$ -ferrocyanide was found to crystallize from a warm satd. soln. of  $\alpha$ -ferrocyanide containing 1% of Hofmann's Na aquopentacyanoferrate. The  $\beta$ -ferrocyanides are, therefore, also mixed crystals containing too little of the ferrate to be detectable by analysis. Since  $\beta$ -ferrocyanide is formed when  $\alpha$ -ferrocyanide is repeatedly recrystd. from water, it follows that ferrocyanides in aq. soln. are in equil. according to the equation  $\text{H}_2\text{O} + \text{K}_4\text{Fe}(\text{CN})_6 \rightleftharpoons \text{K}_3[\text{Fe}(\text{CN})_5\text{H}_2\text{O}] + \text{KCN}$ . Acids increase the velocity of the reaction toward the right, alkalis that toward the left.

A. R. MIDDLETON

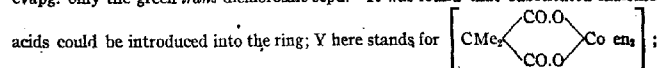
**Complex metallic amines. IV. *Cis*-sulfoacetato-, *cis*-methionato- and *cis*-dimethylmalonato-diethylenediaminecobaltic salts.** T. S. PRICE AND J. C. DUFF. *J. Chem. Soc.* 117, 1071-7(1920); cf. *C. A.* 10, 174, 862; 14, 3024.—The prepn. of these compds. furnishes additional proof of the possibility of rings containing 8 atoms when S is one of these atoms. The bromides of the salts described were obtained in cryst. form by mixing aq. solns. of  $[\text{Co}_2\text{Co en}_2]\text{Br}$  and the acid, evapg. to small vol. and keeping on ice till crystn. took place. The following salts in which Y stands for



brick-red;  $\text{Y}_2\text{PtCl}_6$ , pale brown, microcrysts.;  $\text{YSO}_3\text{CH}_2\text{CO}_2\text{H}$ , pink microcrysts., by addition of 2 mols. sulfoacetic acid to 1 mol. of carbonate hydroxide.  $\text{Y} =$



elongated plates;  $\text{YSCN}$ , dark red microcrysts.;  $\text{Y}_2\text{PtCl}_6$ , light brown; normal methionate  $\text{CH}_3(\text{SO}_2\text{Y})_2$ , pink microcrysts.; the hydrogen methionate could not be obtained. Contrary to expectation neither of these acids yields additive compds. with their acid salts of  $[\text{Cl}_2\text{Co en}_2]$ ; on boiling, the soln. took on the red color of the *cis*-salt but on evapg. only the green *trans*-dichlorosalt sepd. It was found that substituted malonic



$\text{YBr} \cdot 2\text{H}_2\text{O}$ , crimson-red microplates;  $\text{YNO}_3 \cdot 2\text{H}_2\text{O}$ , crimson, microcryst. powder;  $\text{YCl}_3 \cdot 2\text{H}_2\text{O}$ , microcryst. prisms, very sol. in water.

A. R. MIDDLETON

**Action of methyl and ethyl sulfates on alkali phosphates in aqueous solution.** O. BAILLY. *Bull. soc. chim.* 27, 744-50(1920).—See *C. A.* 14, 2452. E. H.

Addition compounds of the acetylacetonates of rare earths (JANTSCH, MEYER) 10.  
Interaction of carbon disulfide with ammonium carbonate (GILFILLAN) 10.

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SPEAR, R. H.: *A Junior Inorganic Chemistry*. London: J. and A. Churchill. 386 pp. 10s. 6d. net. For review see *Nature* 106, 240(1920).

WERNER, A.: *Neuere Anschauungen auf dem Gebiete der anorganischen Chemie*. Die Wissenschaft, Bd. 8. 4th Ed. enlarged. Edited by P. Kadrer. Braunschweig: Friedr. Vieweg & Sohn. 432 pp. M. 18. For review see *Z. physik. Chem.* 96, 252 (1920).

## 7—ANALYTICAL CHEMISTRY

WILLIAM T. HALL

Review of analytical chemistry. ANDRÉ KLING AND ARNOLD LASSIEUR. *Chimie industrie* 4, 151-64, 324-33, 457-66(1920). E. H.

The salt error of cresol red. ROGER C. WELLS. *J. Am. Chem. Soc.* 42, 2160-74 (1920).—The salt error of cresol red, an indicator used in detg. the H-ion concn. in sea water, has been studied so that in making comparisons the proper allowance could be made for the amts. of salt in the standard and the unknown. The results obtained show that with the buffer mixts. employed, there is a considerable difference in the  $p_H$  values for mixts. having the same color in the presence and absence of salts such as NaCl, this difference depending on the concn. of salt and also slightly on the concn. of the buffer. A table is given of corrections for  $p_H$  (marked on the standard) for varying salinities in the unknown. H. JERMAIN CREIGHTON

The volumetric methods for estimating tin. J. G. P. DRUCE. *Chem. News* 121, 173-4(1920).—Volumetric methods for detg. Sn were compared by using a standard  $K_2SnCl_6$  soln. The most convenient volumetric process was the detn. in acid soln. by standard I soln. This is applicable to  $SnCl_2$ , to inorg. and to aliphatic amine chlorostannites (C. A. 12, 1622, and 13, 1061), but is less suitable for aromatic amine chlorostannites owing to the solns. darkening during titration. This method chosen by D. has been previously described (*J. Am. Chem. Soc.* 19, 515 and 807(1897) and *Chem. News* 84, 167(1901)). C. C. DAVIS

Sorption by copper sulfide (separation of copper and zinc). K. SCHERINGA. Utrecht. *Pharm. Weekblad* 57, 1294-5(1920).—The carrying down of sol. salts by  $CuS$  ppts. (C. A. 12, 1522) is a phenomenon which is very sensitive to conditions (especially of temp.) and hence extremely difficult to reproduce quant. in successive expts. This is ascribed to the fact that pptn. consists in the formation of labile substances which, during their transition to stable forms, tend to exclude from the crystals all impurities. The completeness of this exclusion is greatly influenced by external conditions. JULIAN F. SMITH

A rapid qualitative method for nickel in steel. MELVIN B. DANHEISER. *Chem. Met. Eng.* 23, 770(1920).—Place on a watch glass enough filings to cover the head of a pin, add a drop of  $HNO_3$  (d. 1.42) and warm if necessary to start effervescence. In half a min. add drop by drop ammoniacal dimethylglyoxime reagent (5 g. citric acid dissolved in 90 cc. of 1:1  $NH_4OH$  (d. 0.90) with 10 cc. of 1% soln. of dimethylglyoxime in EtOH) until action ceases and the soln. becomes pink. Let stand 1 min., then with a 16 mm. objective examine under the microscope for Ni dimethylglyoxime crystals, massed in pink needles. With no Ni, a brown color forms on standing, with formation of brown crystals when evapd. almost to dryness. C. C. DAVIS

A study of the determination of potassium as the perchlorate, and the separation from sodium, etc. R. LITCH MORRIS. *Analyst* 45, 349-68(1920).—A discussion of the perchlorate method, showing by means of known solns. the errors to be expected. To insure accuracy, a detailed procedure is recommended for general use when Na, Mg, Ca, Fe and Al compds. and citric and tartaric acids are present. M. recommends (1) maintenance of a working temp. of not over 20°, (2) preliminary removal of all  $\text{NH}_4$  compds., (3) 3 evapns. with 20%  $\text{HClO}_4$  soln., (4) dissolving the ppt. and re-evapn. if Na or other bases are present, (5) a wash liquid at least 97% EtOH with 0.2%  $\text{HClO}_4$ , (6) a minimum use of wash liquid, (7) elimination of final washing with pure EtOH by using asbestos filters, (8) the use of a Gooch crucible and (9) drying not below 130°. Procedure: Evap. the soln. 3 times with 20%  $\text{HClO}_4$  soln. to dryness (stop when a paste if Mg compds.,  $\text{H}_3\text{PO}_4$ , or org. acids are present). Treat the residue with 10 cc. of wash liquid, let stand with stirring 15 min. if Na is present, allow to settle and decant through an asbestos Gooch. With K alone present, rinse the ppt. out with wash liquid. When Na is present, evap. the residue from the first decantation to remove EtOH, dissolve in boiling  $\text{H}_2\text{O}$  and evap. to dryness. Treat with 10 cc. of wash liquid, again decant, and transfer with 10 cc. of the filtrate. Wash the filter with a measured amt. of wash liquid. Dry the Gooch 1 hr. at 130-50°, cool and weigh. Wash with 3 cc. more, dry and weigh. Repeat until successive weighings vary not over 0.1 to 0.2 mg. C. C. DAVIS

The determination of the calcium content in the sulfur acids by means of the Feld-Sander method. R. SIEBER. *Svensk Pappers-Tidning* 23, 247(1920).—Because of discrepancies in results for Ca by methods of different investigators critical expts. were made which showed that the method of Feld as simplified by Sander gives the correct results and is not affected by those factors which influence the results of the methods of Winkler and Hoehn. W. SIEGERBLUM

Perchloric acid as a dehydrating agent in the determination of silica. H. H. WILLARD AND W. E. CAKE. *J. Am. Chem. Soc.* 42, 2208-12(1920).—The dihydrate of  $\text{HClO}_4$  boils at 203° and at this temp. is a powerful dehydrating agent. Results are given for the estn. of  $\text{SiO}_2$  in metals and alloys, limestone and sol. silicates showing apparent errors of only a few tenths of a mg. ALBERT SALATHE

A method for determining the sulfur monochloride content of mustard gas- $\text{S}_2\text{Cl}_2$  mixtures. W. A. FELSING, S. B. ARENSEN AND F. J. KOPP. *J. Ind. Eng. Chem.* 12, 1054-6(1920).—In the manufacture of mustard gas from  $\text{S}_2\text{Cl}_2$  and  $\text{C}_2\text{H}_4$ , it is important to control the  $\text{S}_2\text{Cl}_2$  content of the reaction mixture. At Edgewood Arsenal a method of analysis has been devised which is based upon the reaction  $\text{S}_2\text{Cl}_2 + 2\text{NaI} = \text{I}_2 + 2\text{S} + 2\text{NaCl}$ , the liberated I being titrated with standard thiosulfate soln. ALBERT SALATHE

A new method for the estimation of methanol. S. B. SCHRYVER AND CYRIL C. WOOD. *Am. J. Pharm.* 92, 720-8(1920).—See *C. A.* 14, 2601. W. G. GAESSLER

Laboratory notes. Detection of mercury in the presence of bismuth. Standardization of volumetric solutions with cochineal and methyl red as indicators (BOLLINGER, TERRY) 17. Analysis of plating solutions (HAAS) 4. Microchemical reaction for ammonium salts (VAN ZIJF) 11D.

SCHOORL, N.: *Organische analyse. I. Algemeen gedeelte.* 2nd Ed. Amsterdam: D. B. Centen. 156 pp. For review see *Pharm. Weekblad* 57, 1353(1920).



## 8—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHEERRY AND WALTER F. HUNT

The occurrence of *naumannite* in Idaho. E. V. SHANNON. U. S. Nat. Museum. *Am. J. Sci.* 50, 390-1(1920).—The specimen was collected from the silver stopes, DeLamar, Owyhee Co. It is coated with grayish clay but the interior has a dark blue-gray color with metallic luster, is sectile and malleable, and does not tarnish as readily as the sulfide.  $H = 2.5$ , sp. gr. 6.527. The pure mineral does not equal 7.0 in sp. gr. as compared with 8.0 given for *naumannite* in the textbooks. Aside from the clay, the only other mineral present is *marcasite*. Deducting these two impurities, analysis gave: Ag 75.98, Se 22.92, S 1.10, sum 100.00%. Pb, Cu, Au, Zn, Bi, Sb, As and Te were absent. Many tons of this ore were mined, and the Ag-bearing mineral was supposed to be *argentite*. L. W. RIGGS

The nomenclature and classification of sulfide minerals. EDGAR T. WHEERRY. Wash., D. C. *J. Wash. Acad. Sci.* 10, 487-96(1920).—An application of the methods previously used with the elements (*C. A.* 11, 2570) to the sulfides and related compds. including those of Se, Te, As, Sb, Bi, and also O + S, N, P, C, and Si. Variability in compn. is frankly admitted, and shown in the formulas by a dash between the limiting amts. of the constituents; for example, the 1st mineral listed, *wehrlite*, is assigned the formula  $Bi_{1-x}(Te, Se, S)$  to show its widely variable compn., especially as to Bi content. The arrangement is first on the basis of metallic or non-metallic character of the more basic element concerned, and next into chem. divisions, depending on the ratios of the basic to the acidic elements present (giving 3:1, 4:3, 1:1, etc., divisions). These are subdivided into groups, on a crystallographic basis, arranged in the order of decreasing symmetry, the final group in many divisions including amorphous and massive minerals. About 125 minerals, with their formulas, are thus arranged, some of them not yet having received mineralogical names. A number of supposed species are reduced to varieties or mixtures, but some heretofore classed as varieties are raised to specific rank. E. T. W.

*Boulangerite*, *bismutoplagonite* and a silver-bearing variety of *jamesonite*. E. V. SHANNON. *Proc. U. S. Nat. Museum* 58, 589-604(1920); cf. *C. A.* 14, 1505.—(1) Analyses of 14 specimens are given, including Boulanger's original analysis, made in 1835, and 7 new ones by S., the specimens originating in Europe, Asia, N. and S. America. The formula assigned *boulangerite* by Rammsberg in 1875 was  $3PbS.Sb_2S_3$ , although the figures in some cases indicated  $5PbS.2Sb_2S_3$  and in others  $10PbS.3Sb_2S_3$ . Sjögren found the best data to favor  $5PbS.2Sb_2S_3$ . A sample from Mullan, Idaho, analyzed by Shannon (*C. A.* 12, 666) gave  $5PbS.2Sb_2S_3$ , and was named *mullanite*, in oversight of Sjögren's work. Other recent analyses of *boulangerite* give almost exactly the 5:3 ratio, so *mullanite* should be eliminated from the literature. (2) Cf. *C. A.* 14, 1505; the mineral is named *bismutoplagonite* as it falls in line with *plagonite* and *liveingite*, which contain Sb and As, resp., in place of the Bi. (3) A specimen of Ag ore from Silver City, Owyhee Co., Idaho, gave on analysis: Pb 40.77, Cu 0.75, Fe 0.46, Ag 7.40, Sb 30.61, S 20.81, sum 100.80%, from which the ratios indicate approx.  $2PbS.Sb_2S_3$ . Taking the Ag into account and expanding, the formula becomes  $8PbS.2Ag_3S.5Sb_2S_3$ , with Ag in a small measure replaced by Cu. This does not differ greatly from the "*jamesonite*" from Star City, Nevada, considered to be  $2(Pb, Ag, Cu)_3S.Sb_2S_3$ . L. W. RIGGS

Crystal structures of some carbonates of the calcite group. R. W. G. WYCKOFF. *Geophys. Lab. Am. J. Sci.* 50, 317-60(1920).—The object of this study was to learn if the Laue photographs would yield information which would serve to place the O atoms with considerable accuracy. The method used was that of Nishikawa (*C. A.* 9, 888). The exptl. work and results are shown in 17 figures and are discussed mathe-

atically. Conclusions: A probably unique soln. has been obtained for structures of *calcite* and *rhodochrosite*. Of the assumptions commonly made in crystal structure study, the only one required in these detns. was that the atoms reflect X-rays in an amt. roughly proportional to their atomic numbers. The structure of *siderite* is shown to be so nearly the same as that of *rhodochrosite* as to be indistinguishable by the means at hand. *Magnesite* is shown to give the same sort of pattern, and hence to have the same general arrangement of atoms, as the other members of the group. The positions of the O atoms as indicated by the present method and by spectrometer results are compared. The "normal" decline of intensities is in surprising agreement with the reflections. The use of gnomonic projections in studying Laue photographs is mentioned and a *ruler* is described, the use of which reduces the time and labor of making such projections. The effect of the voltage impressed on the X-ray tube upon the character of the Laue photograph is considered and the *best conditions for operating a W tube* for this work are stated. A criterion is suggested for detg., in the case of a hexagonal crystal, whether the fundamental unit is a rhombohedron or hexagonal prism. Evidence is obtained from these crystal structures to show the existence of groups of atoms, as carbonate groups, in the crystal. It is pointed out that unless every atom in the crystal is electrostatically charged, the outside electrons of the atoms making up these crystals cannot be arranged at the corners of cubes. Some connections are pointed out between the development of faces on calcite and its crystal structure, and a means is indicated of deciding the most probable indices of a plane, when they are in doubt. The bearing of these structures upon the question of what constitutes a series of isomorphous substances is mentioned.

L. W. RIGGS

Existence of thortveitite, a silicate of scandium and yttrium, in Madagascar. A. LACROIX. *Compt. rend.* 171, 421-3 (1920).—Wilkite, which has been used as a source of Sc, contains but 1.2%. Schetelig (*C. A.* 6, 726) has described thortveitite from Norway, with the compn.  $(\text{Sc}, \text{Y})_2\text{Si}_2\text{O}_7$ . Among specimens of pegmatite from Befanamo, east of Ankazolie, Madagascar, was noted an unknown mineral with the physical properties of thortveitite. Spectroscopic examn. showed Sc most marked, also Y, Yb, and indications of Zr, Al and Ti, establishing the identification.

L. W. RIGGS

Analyses and optical properties of amesite and corundophilite from Chester, Mass., and of chromium-bearing chlorites from California and Wyoming. E. V. SHANNON. *Proc. U. S. Nat. Museum* 58, 371-9 (1920).—(1) A specimen from the emery mine at Chester, Mass., consisted of a large mass of diasporite containing amesite in tabular crystals. This has been described (*C. A.* 14, 915), but errors in the first article are corrected and additional data given. Its *ns.* are:  $\alpha = 1.597$ ,  $\beta = 1.597$ ,  $\gamma = 1.612$ . (2) A specimen of corundophilite from the emery mine at Chester, occurs in crystals, granular, and schistose. Color greenish black, and  $\alpha = 1.600$ ,  $\beta = 1.603$ ,  $\gamma = 1.610$ . It is optically +, biaxial,  $2E = 60^\circ$ , with the bisectrix inclined at about  $10^\circ$ , these last 2 points readily distinguishing it from amesite. Analysis gave:  $\text{SiO}_2$  23.20,  $\text{Al}_2\text{O}_3$  24.42,  $\text{Fe}_2\text{O}_3$  3.48,  $\text{FeO}$  13.40,  $\text{CaO}$  1.04,  $\text{MgO}$  22.76,  $\text{H}_2\text{O}$  12.00, sum 100.03%; formula  $6(\text{Fe}, \text{Mg})0.2(\text{Al}, \text{Fe})_2\text{O}_3.3\text{SiO}_2.5\text{H}_2\text{O}$ . (3) A specimen from the mine of the Placer Chrome Co., Newcastle, Cal., shows coatings of a minutely scaly material of pale lavender color associated with unarovite on granular chromite. Analysis gave:  $\text{SiO}_2$  29.36,  $\text{Al}_2\text{O}_3$  18.81,  $\text{Cr}_2\text{O}_3$  1.53,  $\text{MgO}$  35.67,  $\text{CaO}$  2.20,  $\text{FeO}$  1.65,  $\text{H}_2\text{O}$  11.34, sum 100.56%; formula  $10\text{RO}.2\text{R}_2\text{O}_3.5\text{SiO}_2.6\text{H}_2\text{O}$ . The optical character is +, with  $\alpha = 1.582$ ,  $\beta = 1.582$ ,  $\gamma = 1.593$ . (4) A sample from Deer Park, Wyo., consisted of granular chromite-containing veins and disseminated crystals of deep red chlorite. By hand picking a sample was obtained which contained chromite only as minute grains between the folia of the chlorite. Analysis gave: chromite 2.04,  $\text{SiO}_2$  32.12,  $\text{Al}_2\text{O}_3$  9.50,  $\text{FeO}$  1.98,  $\text{Cr}_2\text{O}_3$  7.88,  $\text{MgO}$  35.36,  $\text{CaO}$  1.24,  $\text{H}_2\text{O}$  10.25, sum 100.37%; formula  $6\text{RO}.2\text{R}_2\text{O}_3.4\text{SiO}_2.4\text{H}_2\text{O}$ . The mineral is biaxial, with a very small

optic angle and having the acute bisectrix normal to the perfect cleavage:  $\alpha = 1.587$ ,  $\beta = 1.590$ ,  $\gamma = 1.590$ . Under the existing classifications both the Cal. and Wyo. Cr-bearing chlorites would be referred to clinocllore or to the Cr-bearing varieties *kammererite* and *kotschubeite*.

L. W. RIGGS

**Chemical composition of Post-Tertiary clay material.** O. TAMM. *Geol. För. Förh.* 41, 462-8(1919).—Analyses of clay from 4 different deposits in Sweden showed distinctly lower  $\text{SiO}_2$  and higher Fe and Mg content than in the coarser layers. K. was often higher, Na lower, and Ca content variable. The Al content was always noticeably higher than the max. for the kaolinite formula, from 5 up to 12%. W. S.

**Mineral resources of Alaska—report 1918.** G. C. MARTIN, *et al.* U. S. Geol. Survey, *Bull.* 712, 198 pp.(1920).—The administrative and statistical reports fill 41 pages. A falling off in production is noted on account of scarcity of labor and materials, except in the production of coal, which is increased 40% over 1917. A slight increase in petroleum is noted. An event of much interest was the recovery of substantial amts. of Pd and some Pt from the Cu ores of Salt Chuck mine near Ketchikan. Water power investigations in S. E. Alaska. G. H. CANFIELD. *Ibid* 53-90.—**Nickel deposits in the lower Copper River valley.** R. M. OVERBECK. *Ibid* 91-8.—Spirit Mt. Ni deposits are near the head of Canyon Creek and not far from mile post 121 on the Copper River and N. W. R. R. Analysis of the ore give Ni ranging from a few % up to 19.6. Preliminary report on the chromite of Kenai Peninsula. A. C. GILL. *Ibid* 99-129. Cf. C. A. 14, 2457. Mining developments in the Matanuska Coal Field. T. CHAPIN. *Ibid* 131-68.—These fields are located in the valley of the Matanuska River, a tributary of Cook Inlet, 50 to 75 miles from the town of Anchorage. The areas of supposed coal-bearing rocks cover 70.3 sq. mi. with possible extensions of 25 sq. mi. Analysis and steaming tests show the coal to be "suitable in every respect for use in the naval service." The coal is described as ranging from high-grade to low-grade bituminous and some of it appears to have good coking qualities. The production of this field in 1918 was 63,000 tons. Four mines are described, the Chickaloon and Eska mines with much detail. The development of these mines has continued since 1918 with most promising indications. Lode developments in the Willow Creek District. T. CHAPIN. *Ibid* 169-76.—Ten mines or prospects are briefly described. The production for 1918 was Au \$270,000, Ag \$725. Placer mining in the Tolovana District. R. M. OVERBECK. *Ibid* 177-84.—The output of this district on 1918 was Au \$875,000 from placers in the vicinity of Livengood. Ores of Cr, W, Sb, and perhaps Pt occur but have not been developed sufficiently to est. their possibilities. Mining in Northwestern Alaska. S. H. CATHCART. *Ibid* 185-98.—Various climatic and economic factors combined to lower the output of the mines of the Seward Peninsula to \$1,195,000 in 1918, or less than one-half of the value of the year 1917. Of this total \$87,172 represents Sn, W, Ag, and Pt, the remainder Au. Expts. were made in thawing the ice out of frozen ground by means of superheated steam, hot water, and water a few degrees above the f. p.

L. W. RIGGS

**Copper deposits of Lake Superior.** S. S. LANG. *Mining Sci. Press* 121, 407-8 (1920).—The Lake Superior deposits were formed by heat emanating from local intrusives, causing intense circulation of ascending solns., forming a secondary concn. of rich Cu ores from the original very low grade or lean zeolitic deposits in the basaltic flows.

S. G. GORDON

**Gypsum deposits of the United States.** R. W. STONE, *et al.* U. S. Geol. Survey, *Bull.* 697, 316 pp.(1920).—The production of gypsum in the U. S. increased over 300% during the 14 years ending with 1918, the production for that year being valued at \$11,000,000. The mineralogical and chem. features of gypsum in its many varieties are described and 52 analyses are quoted. The liability of error in the chem. analysis of gypsum is pointed out by George Steiger, who has reviewed the analyses of several

chemists and showed that the water of hydration given is variable, depending on the humidity of the air and the state of division of the mineral. The history, production by states, imports, technology of mining and grinding and uses of gypsum are given. The development of the gypsum deposits and industries in 24 states is described, either by Stone or by the State Geologist of the respective state. L. W. RIGGS

Beerbachite at the Lizard. T. G. BONNEY. *Geol. Mag.* 57, 339-40(1920).—Microscopic examn. showed this rock to consist of hornblende, augite, labradorite, and Fe oxides. S. G. GORDON

Petrographic studies in the Nyberg district. I. HÖGBOM. *Geol. Förr. Förrh.* 42, 105-74(1920).—This paper includes 11 photomicrographs of mineral sections and 9 tables of chem. analyses of leptite rocks found in the Swedish Fe fields. These analyses are used in trying to find a common theory for the genesis of Fe ores, without complete success. W. SEGERBLOM

Chemical changes during metamorphism. H. BACKLUND. *Geol. Förr. Förrh.* 41, 383-95(1919).—B. gives 2 tables of analyses of granite and of Ca minerals, with a rearrangement of data to bring out the changes which occur. W. SEGERBLOM

The origin of flint. R. M. BRYDENE. *Geol. Mag.* 57, 401-4(1920).—There are 3 kinds of flint: separate, interstratified continuous lines, and contrastratified continuous lines (veins). The origin of these is discussed, a penesimultaneous origin being advocated for the first 2, and a later origin for the veins. S. G. GORDON

The examination of some rocks in the vicinity of Nascente and of the thermal waters of Valadares do Minho (Monsão). V. SOUZA-BRANDAO. *Rev. chim. pura applicada* 4, 129-36(1919).—The eruptive rocks of this region consist mainly of granodiorites through which run dikes of feldspathic mica-schists formed by the metasomatic action of thermal waters. C. B. SLAWSON

Catalog of Swedish geological, paleontological, petrographic, and mineralogical literature for 1907-1917. FR. E. ÅHLANDER. *Geol. Förr. Förrh.* 41, 539-606(1919).—A list of titles, names of authors and references for about 1000 papers, arranged alphabetically by authors, published in Sweden and elsewhere. Many are in English, and some are chem. in nature. W. SEGERBLOM

Constitution of silicates (JAKOB) 6. Magnesium (VICKERS) 4. The fluorine content of mineral waters (CARLES) 14. The fluorine in the mineral waters of Portugal and Spain (FERRERA DA SILVA, D'AGUILAR) 14.

GOLDSCHMIDT, VICTOR: Beiträge zur Krystallographie und Mineralogie. Vol. I. Heidelberg: Carl Winters Universitäts Buchhandlung. 264 pp. For review see *Am. J. Sci.* 50, 398(1920).

LACROIX, A.: Les industries minérales non métallifères a Madagascar. Paris: Editions de la Revue politique et littéraire.

## 9—METALLURGY AND METALLOGRAPHY

WILLIAM BRADY, ROBERT S. WILLIAMS

Testing ores for flotation. JAMES M. HYDE. *Mining Sci. Press* 121, 481-6 (1920).—The factors involved in the concn. of ores by flotation are so few in number and so readily applied that it is possible to gain a fairly accurate idea of the character of the concentrate that may be obtained by flotation and even the total percentage of recovery that may be made by this process by means of simple app. and small quantities of ore. In detg. the general nature of the concentrate obtainable from a given ore a mixt. of oils and tars which are generally applicable is useful. Such a mixt.

is 3 parts pine creosote, 3 parts carbolic creosote and 1 part crude coal tar. The simplest app. for testing ores is a clear glass bottle. In using this the shaking is done by hand and the concentrate overflowed by adding water beneath the surface by means of a funnel. A number of testing machines are described in detail. R. S. DEAN

The effect of addition agents in flotation. II. MARTIN H. THORNBERRY AND HORACE T. MANN. *Bull. School Mines and Met. Univ. Mo.* 1919, Pt. 2, 65 pp.—The effect of the addition of chlorides, acetates, oxalates, chromates and other agents on the flotation of a southwest Missouri lead ore was detd. The oils used were Cleveland Cliffs Iron Co.'s flotation oil No. 1 (hardwork creosote), General Naval Stores flotation oil No. 17 and cresylic acid. The results with the 3 oils were similar. The expts. show that all the addition agents lower to a slight extent the extn. and the grade of concentrate; the following substances were, however, found particularly deleterious  $\text{SnCl}_4$ ,  $\text{UCl}_4$ ,  $\text{VdCl}_3$ ,  $\text{UO}(\text{OAc})_2$ ,  $\text{Cd}(\text{OAc})_2$ ,  $\text{H}_2\text{CrO}_4$ ,  $\text{K}_2\text{CrO}_4$ ,  $\text{Na}_2\text{CrO}_4$ ,  $\text{KMnO}_4$ , tannic acid, tartaric acid,  $\text{CdBr}_2$ ,  $\text{UBr}_4$ . Altogether some 85 substances were tried. R. S. DEAN

The application of the Bradford flotation process to mixed sulfide concentrates. W. D. GREEN AND WM. FAGERGREN. *Mining Sci. Press* 121, 455-8(1920).—The selective flotation of lead and zinc in mixed sulfide concentrates made from the tailings of the U. S. Smelting Co. plant at Midvale, Utah, could not be accomplished with uniform success following the Australian practice of using either acid and  $\text{Na}_2\text{S}_2\text{O}_4$  or acid and  $\text{SO}_2$ . It was found that satisfactory results could be obtained using  $\text{SO}_2$  alone. The plant was later operated on tailings from the U. S. mill. Details of the mill are given. R. S. DEAN

Shattuck Arizona mill for concentrating silver lead carbonate ores. GLENN L. ALLEN. *Eng. Mining J.* 110, 759-62(1920).—The ore contains Au 0.06 oz., Ag 5 oz., Pb 5.3%, Fe in hematite and limonite 13%, insol. 71%, S less than 0.1%,  $\text{CaO}$ ,  $\text{Al}_2\text{O}_3$  and Mn, less than 1%. The ore is oxidized and low-grade. A 400-ton mill handles 500 tons in 24 hours. Details of crushing, gravity concn. and flotation are given. The slimes, 92% through 200 mesh, are sulfidized in the thickener by the application of com. 60% sodium sulfide at the rate of 3 lbs. per ton of ore. The solids form 20 to 30% of the slimes. The treatment forms  $\text{PbS}$  and  $\text{Ag}_2\text{S}$ . The pulp is then well oiled in an agitator by treatment successively with a mixt. of coal-tar and hard-wood creosotes and later with a similar mixt. containing oil-tar and some times a little pine or eucalyptus oil. K. & K. and Rork flotation cells are used. The recoveries are Au 69.4%, Ag 71.3%, Pb 94.4%. J. O. HANDY

Progress in ore dressing and coal washing in 1919. ROBERT H. RICHARDS AND CHARLES E. LOCKE. *Mineral Ind.* 28, 747-801(1919).—An annual review, covering crushing and grinding, screening, classifying, settling, jigs and tables, amalgamation, magnetic and electrostatic processes, flotation, and treatment of coal, with examples of practice and a complete bibliography for the year. A. B.

Iron and steel. EDWIN F. CONE. *Mineral Ind.* 28, 337-92(1919).—Statistics and review of the world's production and trade, including ore, pig iron, steel, electric furnaces, markets, and technology. A. B.

Lead. H. B. PULSIFER. *Mineral Ind.* 28, 393-417(1919).—Statistics of Pb and Pb products are given, covering the world's production and market. A. B.

Recent improvements in lead smelting. H. O. HOFMAN. *Mineral Ind.* 28, 418-32(1919).—A review of progress in Pb metallurgy. A. B.

Manganese. MARSHALL HANEY. *Mineral Ind.* 28, 442-60(1919).—A review of the industry, domestic and foreign. A. B.

Molybdenum. ALFRED W. G. WILSON. *Mineral Ind.* 28, 467-75(1919).—Data on the world's production, with notes on the market and uses. A. B.

Nickel. THOS. W. GIBSON. *Mineral Ind.* 28, 479-88(1919).—Deposits, metallurgy and production are discussed, with statistics of output and trade. A. B.

**Platinum.** GEORGE F. KUNZ. *Mineral Ind.* 28, 549-67(1919).—A review of the world's output and market. A. B.

**Quicksilver.** W. D. BURCHAM. *Mineral Ind.* 28, 814-21(1919).—A review of the industry, domestic and foreign. A. B.

**Tin.** BALIOL SCOTT. *Mineral Ind.* 28, 684-84(1919).—A survey of the world's sources and production of Sn. A. B.

**Tungsten.** COLIN G. FINE. *Mineral Ind.* 28, 688-709(1919).—A review of the industry in the United States and foreign countries, with notes on technology and a bibliography. A. B.

**Uranium and vanadium.** ROBERT M. KEENEY. *Mineral Ind.* 28, 710-9(1919).—Production, market, deposits, and metallurgy are treated. A. B.

**Zinc.** JESSE A. ZOOK. *Mineral Ind.* 28, 720-46(1919).—Statistics of the world's production, market and trade. A. B.

**Metallurgy of zinc.** W. R. INGALLS. *Mineral Ind.* 28, 743-6(1919).—Annual review of new developments. A. B.

**New deoxidizers for steel manufacture.** J. R. CAIN. *Chem. Met. Eng.* 23, 879-82(1920); cf. C. A. 14, 521, 1513.—A report of cooperative investigation work sponsored by the Natl. Research Council on the action and systematic selection of new deoxidizing alloys. Mode of action and general functions of steel deoxidizers are given. The most important function is the removal of solid oxides contaminating the metal, accomplished by formation of deoxidizing slags. The most fusible deoxidizing slags are best. The main object of these investigations was to det. the most fusible oxide combinations, from which the compn. of corresponding deoxidizing alloys might be calcd. The work is limited to the elements Mn, Si, Al and Ti, and further confined to binary and ternary systems. Ferrous oxide was not used in the exptl. work, but its presence in actual practice makes for a lower fusing point of the slag. In the exptl. work Armco iron crucibles, m. 1500°, were used, and the work was carried on in a non-oxidizing atm. The U. S. Bur. of Standards' method of detg. fusing point is given. Seventy-three deoxidizing alloys were found having m. p., when oxidized, below that of pure Fe. Of the new deoxidizers the most fusible found were (a) 75% Mn, 24% Si, m. 1228°; (b) 41% Mn, 59% Ti, m. 1160°; (c) 49% Mn, 18% Ti, 33% Si, m. 1130°; (d) 13% Al, 52% Mn, 35% Si, m. 1121°; (e) 12% Al, 47% Mn, 41% Ti, m. 1320°.

A. H. HELLER

**An unusual process in the treatment of gold ore.** B. L. GARDINER. *J. Chem. Met. Mining Soc. S. Africa* 21, 9-11(1920); cf. C. A. 14, 3210.—Reply to discussion. At Connemara there is no evidence that low Au extn. without the baking process was due to reducing compds. such as Fe<sup>++</sup>, either in the ore or in the soln. A. BUTTS

**United Verde smelter.** L. A. PARSONS. *Mining Sci. Press* 121, 545-57(1920).—The smelter, located at Clarkdale, Ariz., is producing 7,000,000 lbs. of Cu from 74,000 tons of ore monthly; 8.5 tons of charge are being smelted per sq. ft. of blast-furnace hearth. A 25.7% mat is made from 5.87% ore with a remarkably low coke consumption. The operating costs in January were \$1.84 per ton for blast-furnace operation, \$2.13 per t. for reverberatory, \$0.23 for Wedge-furnace roasting, \$12.00 per ton for blister copper for the converter. Recovery is 88.54% and will be improved by a Cottrell dust precipitator. The smelter capacity is to be made 15,000,000 lbs. Cu per month. 8400 boiler h. p. are to be generated from waste heat. Coal-dust firing is to displace oil fuel in reverberatories. The chem. and mineralogical composition of the ores is given. The roaster, blast-furnace and converter plants are described in detail. So also are the coal-pulverizing plant, the power plant and the machine shop.

J. O. HANDY

**Roasting and chloridizing of Bolivian silver-tin ores.** M. G. F. SÖHNLEIN. *Mining and Met.* 1920, No. 164, 21-2; *Mining Sci. Press* 121, 384-6(1920).—The ores

in question consist of tetrahedrite, jamesonite, stibnite and cylindrite together with cassiterite in a gang of quartz and pyrite. Treatment includes dry crushing, a chloridizing roast, leaching with water and  $\text{Na}_2\text{S}_2\text{O}_3$  soln. to extract Au, Ag and Cu, followed by concn. of Sn. Rise in labor costs made necessary the substitution of the McDougall furnace for the hand-rabbed reverberatory, but stack losses of Ag rose from 6% or less to considerably more than 20%, varying with the percentage of S and attendant rise of furnace temps. At present Ag losses are reduced somewhat by charging the McDougall with low-grade fines of run-of-mine ore of high S content mixed with Ag-Sn ore carrying only 15%-20% S. The objection to the use of the Cottrell app. is (1) difficulty of installing it above the furnace, and (2) of disposing of the dust which consists of raw, half-oxidized and oxidized ore. The best solution of the disposal problem would be treatment by a hydro-metallurgical process. H. L. OLIN

Core baking in electrically heated ovens. JESSIE L. JONES. *Metal Ind.* 18, 450-1 (1920).—Details of construction of the furnace and thermo-regulator, capable of holding within 5° any temp. between 150 and 1450° F., are not given. Comparative tests were made between the electrically heated oven and others heated with gas or oil. The binder was a mixture of linseed and mineral oils with or without dextrin. The cores were baked at 300 to 450° F. The results obtained were in favor of the elec. furnace as follows: (1) Oven temp. uniform throughout; (2) temp. held steady within 5°; (3) no annoyance from fuel combustion products; (4) cores well baked in one hour at 450° F.; (5) cores 50 to 200% stronger, when tested by compression; (6) cores may be baked much faster; (7) about 1 kw.-hr., of elec. power required for 12 lb. of green cores. J. O. HANDY

From a metallographic practice. M. V. SCHWARZ. *Z. Metallkunde* 12, 1-27 (1920).—The paper deals with a number of investigations carried on during the early part of the war and is concerned chiefly with the manuf. of munitions. Section I shows by means of a series of micrographs and a hardness table the connection between the amt. of cold work on brass shell cases and the Brinell hardness. It deals also with the hardening of Zn-Al alloy. II deals with the rolling of steel shell cases and shows by a series of tensile-strength tables and photomicrographs the advantages of shells made from rolled material over those pressed directly from the ingot. III discusses the methods of hardening the inner surfaces of iron tubes. IV is a series of investigations on cold-rolled shells of different sorts of steel. Tables are given showing the physical properties of different samples in sheet form and the difficulties both with the finished shells and the rolled sheets from which they were made are illustrated by an excellent series of S prints and macrographs. V deals with the methods of photomacrography. VI outlines a series of expts. on the determination of the correct rolling and working temperature of a Zn-rich alloy containing small amounts of Pb and Cu. Many photographs of fractures at different working temps. are given. R. S. WILLIAMS

Macrography in the industries. E. VIGNE. *L'Age de fer* 36, 433-5 (1920).—A summary of the methods of macrographic study of the alloys is given including the methods of polishing, etching by means of various reagents and interpreting the results of low-power examn. of alloys. Sketches are given to illustrate the applications of the methods to various types of engineering alloys. R. S. W.

Determination of the specifications of steel. E. LIÉVENIE. *L'Age de fer* 36, 558-9 (1920).—L. makes a plea for the codification of the specifications of steel on the basis of modern metallographic knowledge. R. S. W.

Physical properties of arc-fused steel. HENRY S. RAWDON, EDWARD C. GROSS-BECK and LOUIS JORDAN. *Chem. Met. Eng.* 23, 677-84 (1920).—Two types of electrodes were used as material to be fused, "A" being representative of a "pure" iron and "B"

that of a low-C steel. The general effect of the fusion was to render the 2 materials used for welding pencils more nearly the same compn. The loss of C and Si was very marked in each case where these elements exist in considerable amts. The most noticeable change in compn. was found to be the increase in the N content of the metal. In general, the % of N taken up by the fused Fe increases somewhat as the current density increases. The metal resulting from the arc-fusion was found to contain a considerable number of cavities and oxide inclusions: these were best seen after the surfaces were etched with a 10% aqueous soln. of  $\text{Cu-NH}_4\text{Cl}$ . The very low ductility shown by specimens when stressed in tension was found to be the most striking feature observed in the mechanical properties of the material as revealed by the tension test. The measured elongation of the tension specimen does not truly indicate a property of the metal. Due to unsoundness, the true properties of the metal are not revealed by the tension test to any considerable extent. The test measures, largely for each particular specimen, the adhesion between the successively added layers, which value varies considerably in different specimens due to the unsoundness caused by imperfect fusion, oxide and other inclusions, tiny enclosed cavities and similar undesirable features. The elongation measured for any particular specimen is due largely, if not entirely, to the increase of length due to the combined effect of the numerous tiny imperfections which exist throughout the sample. That the metal is inherently ductile is shown by the behavior upon bending in the microstructure of bent specimens. The formation of slip-bands within the ferrite grains to the extent which was observed is evidence of a high degree of ductility. The grosser imperfections, however, are sufficient to prevent any accurate measurement of the real mechanical properties of the metal. The changes of chemical compn. which the fusion entails, together with the unusual features of microstructure which accompany the compn. change are of but minor importance in detg. the strength, durability and other properties of the arc weld. The two types of electrodes "A" and "B" should give similar results in practical welding. This is due to the changes which occur during the melting so that the resulting fusions are essentially of the same compn. The use of a slight coating on the electrodes does not appear to be of any material advantage so far as the properties of the resulting fused metal are concerned. The properties of the fused metal are independent of the kind of current and are influenced primarily by the heat of fusion.

V. O. HOMERBERG

Practical notes on the design and treatment of steel castings. GEORGE F. PRES-  
TON. *Chem. Met. Eng.* 23, 529-32(1920).—The foundry practices necessary to eliminate cracks, draws, honeycombing and cavities are discussed. Proper heat-treatment is emphasized, since correct methods will give castings equal to forgings in physical properties.

V. O. HOMERBERG

Colloidal state in metals and alloys. JEROME ALEXANDER. *Trans. Am. Inst. Mining Met. Eng.* 1920 (advance copy), 20 pp.—The object of this paper is to show that many of the important phenomena of metals and alloys are due to the facts that, at some stage, metals and alloys or some of their constituents are in a colloidal state, and that a portion of the metal or alloy tends to remain in this state. The so-called amorphous phase in pure metals is an isocolloid, consisting of colloidal groups of metal molecules that may be dispersed in still more finely subdivided metal; these colloidal particles are not truly amorphous, but may consist, in part, if not entirely, of ultra-microscopic crystals; as a result the expressions amorphous phase and amorphous theory might be replaced by the expressions colloidal phase and colloidal theory. The number and size of the molecular groups in a fused metal tend to increase as the thermal agitation decreases and the solidification point is approached. The higher the temperature to which the metal is heated and the more quickly it traverses the zone of intense thermal agitation to the comparative quiet of solidification, the smaller and



more numerous will be the mol. groups. Pure metals prior to visible crystn. are iso-colloids, but notwithstanding the high viscosity, the powerful crystn. forces act so rapidly that, as a general rule, the major portion of the metal forms macroscopic or microscopic crystals, which are embedded in an intercryst. network of, mainly, colloidally dispersed metal having different physical properties from the crystals. Quick cooling yields fine structure because there is then less opportunity for crystals to grow beyond the colloidal stage; this fact may be demonstrated ultramicroscopically with soaps and glasses. The extent to which the colloidal magma is altered in the direction of a visibly crystalline mass, embedded in a colloidal matrix, is also influenced by the compn. of the phases and the presence of substances that hasten or inhibit crystn. During recrystn. below the m. p. the amorphous or colloidal phase acts as a "solvent" surrounding and nourishing the larger crystals, which tend to grow at the expense of the smaller ones. The effect of colloids on crystn. is very powerful, preventing crystal growth beyond colloidal dimensions, or forcing the crystals to assume globulitic or denticritic forms. This idea is not limited to the field of metallurgy, but applies to any substance or system where a portion or a component is or becomes colloidal while the remaining portion or components are outside of the colloidal zone; the colloid exerts a powerful effect on structure and properties, and may be retained in the final product by adsorption, increase in viscosity, etc. Tin-lead alloys and brasses are discussed from this point of view, also iron and steel. Between 900° and 780° iron is probably an allocolloid, the so-called  $\beta$ -iron being  $\alpha$ - $\gamma$ -iron adsorption compound ( $\alpha$ -iron dispersed in  $\gamma$ -iron) whose decomposition is responsible for the thermal arrest point  $A_2$ . With hypoeutectoid steel the evolution of heat at  $A_1$  seems to be consequent upon the breaking up of an adsorption compd. between  $\gamma$ -iron and iron carbide, the former transforming into  $\alpha$ -iron and the latter being set free to form aggregations of its own. This adsorption compd. explains the carrying over of some  $\gamma$ -iron through the so-called  $\beta$ -iron zone to the eutectoid point, where, with increasing C content, increasing quantities of heat are evolved. With steel of eutectoid compn. (about 0.9% C = 13.5%  $Fe_3C$ ), the large percentage of  $Fe_3C$  carries over practically all the  $\gamma$ -iron in metastable condition to the eutectoid point. The degree of dispersion of the cementite ( $Fe_3C$ ) depends mainly on thermal history and incidental impurities or additions to the steel. While in austenite it might be in true soln., in hardenite and martensite it is colloidal; with troostite it begins to coagulate and pass out of the colloidal zone, and with sorbite and pearlite the coagulation is more marked, being microscopically visible with pearlite. Rosenhain's amorphous theory stresses absence of crystn. or orientation in the amorphous phase, but the X-ray spectroscopy shows that even amicroons in gold hydrosols are cryst. The facts are in harmony with the conception of amorphous metal as an iso-colloid, that is as a dispersion of colloidal crystals, crystal fragments, and non-cryst. groups, forming a solid gel-like mass. The fineness of its particles, and the enormous development of free surface found in all colloids are its critical factors.

JEROME ALEXANDER

Some physical properties of nickel-iron alloys. L. R. INGERSOLL, *et al.* *Phys. Rev.* 16, 126-32(1920).—The results of the investigation showed that the 35% Ni alloy, corresponding to the compn.  $Fe_3Ni$ , has physical properties more or less markedly different from the other ferro-nickels. When the measurements of these physical properties were plotted as a function of the compn. the following general facts were brought out; the m. p. curve shows a max., specific heat a max., thermal cond. a min., thermoelectric power a min., specific resistance a max. and temp. coeff. of resistance a min., all at or very near the compn. 35% Ni.

V. O. HOMERBERG

Alloys of uranium. E. POLUSHKIN. *Rev. Metal* 17, 421-37(1920).—(1) U does not affect the position of  $A_1$  until the content is 7%; then it suppresses all the critical points.  $A_2$  is not visibly lowered until the % of U is more than 2. In steels of less

% it is impossible to see any regular influence due to the U. In steels containing more than 2% U Ar<sub>1</sub> is lowered and gradually suppressed. (2) U exists in steels as oxides and carbides (UC). Under the microscope the oxide is dark gray, slightly bluish. The carbide is recognized by the thermic coloration (method of Stead). It takes on under this influence successively characteristic colors of blue, green and violet. If heating is prolonged the grains of carbide become bluish gray. The carbide is harder, and well crystd. and often contains white crystals. It tends to produce small zig-zag laminations of pearlite. If the % U is high, the pearlite is dark and the laminations are badly formed. (3) UC does not enter into solid soln. with Fe even at 1250°. (4) In steel of av. C content (0.25–0.45%) U increases the elastic limit and modulus of rupture without decreasing the ductility, often the ductility is increased. In steel of 0.60% C, U increases the elastic limit and modulus of rupture, but ductility is considerably lowered. (5) U increases hardness. (6) A casting of Ni-U steel gave good results. It had a ductility greater than ordinary Ni or Cr-V steels, but other castings did not show any favorable effects due to the U. (7) Comparative values of the good effects of U mentioned have not been exactly detd. in view of the special condition of manuf. and heat treatment, but in all cases no extraordinary results have been obtained that cannot be obtained in other special steels. (8) Other special alloy steels have not shown any improvement due to the presence of U. (9) U has no influence on resistance to shock or to alternative tension. E. E. R.

Constitution of certain tin-bearing brasses. W. CAMPBELL. *Am. Soc. Testing Materials* June 1920 (preprint); *Chem. Met. Eng.* 22, 1191(1920).—A relatively slight variation in Sn often causes the most profound physical changes in the alloys. Microscopically, some naval brass castings were found to resemble Muntz metal, in that they consisted of a mixt. of  $\alpha$  and  $\beta$  constituents; some of them appeared like bronzes ( $\alpha$  and eutectoid), while a third class consisted of  $\alpha$ ,  $\beta$ , and eutectoid. All of them were cast to the following specifications: Cu 60 to 65%, Sn 0.5 to 1.5%, Fe 0.5% max., Al 0.5%, Zn remainder. C. studied the region of the ternary diagram, which is included, both as cast and after annealing for 2 hrs. at 650° and furnace-cooled, and obtained results very similar to those of Hudson and Jones. The  $\alpha$ - $\beta$ -eutectoid complex is found in an approx. rectangular area, from which it can be seen that as the Cu content of the cast alloy increases from 56.5%, the amt. of Sn which will cause the alloy to change from the ductile "naval brass" type to the less desirable "gun-metal" type decreases from 2% to less than 0.5% with 67% of Cu. Consequently in proportioning such alloys it should always be clearly understood that the amt. of Sn should be inversely proportional to the Cu. V. O. HOMERBERG

Failure of "manganese bronze." J. H. S. DICKENSON. *Engineering* 110, 556-8 (1920).—See *C. A.* 14, 3212. E. H.

Experiments in manufacture of No. 12 alloy. ROBERT J. ANDERSON. *Chem. Met. Eng.* 23, 883-7(1920).—Expts. were carried on at Gloekler Foundry Co., Pittsburgh, Pa., to obtain comparative data on the manuf. of light Al:Cu alloys by various com. methods. Test methods included keeping variables const., use of plumbago crucibles, of gas-fired melting furnace, and of castings made of each heat, from which microsections were taken. Observations were made as to gas consumption, temp., time and losses. The initial temp. of the crucible was kept as close as possible to 600°. Dross losses and gas consumption were high where Cu or rich alloys were added first and Al later. The reverse was true where the entire charge was melted at once or where rich alloy or Cu was added to the liquid bath. No advantage was gained by adding solid rich alloy or Cu to liquid Al as compared with charging all materials together. In general high dross loss is a result of high temp. Microsections of castings showed no difference as regards microstructure of CuAl<sub>3</sub>, etc., but blow holes and porosity were more numerous on high-temp. melts and where dross loss was high the

following conclusions are reached: (1) One rich alloy is not better than another for introducing Cu, from the standpoint of gas consumption and dross loss. (2) Small heats may be made cheaply by adding light gage Cu to liquid Al; the alloy is formed at low temp. (3) In large-scale production it is safer to employ a rich alloy. (4) The most convenient rich alloy available is the 50:50 alloy. (5) The most desirable method is to charge all materials together; there is no advantage in any other procedure.

A. H. HELLER

Diffusion of solid copper in liquid aluminium. ROBERT J. ANDERSON. *Chem. Met. Eng.* 23, 575-7(1920).—Cu readily alloys with Al when solid Cu is placed in contact with liquid Al. The rate of diffusion is a function of the temp., of the size and shape of the immersed Cu, and of the ratio of the surface area of the solid Cu exposed to the liquid Al. Small pieces of Cu go into soln. in Al fairly rapidly at 700° but very rapidly at 900°. The commercial method for the manuf. of No. 12 alloy by adding Cu directly to Al is a rational procedure from a metallurgical standpoint.

V. O. HOMERBERG

Study of transition points by a dilatometric method (BRAESCO) 2. Industrial readjustments of certain mineral industries affected by the war (RIDDELL, *et. al.*) 13. Ether apparatus and stray electric currents (SCHAAL) 10.

LANDBERG, P.: *Ijzer en Staal*. Amsterdam: Vakbibliotheek onder leiding van L. Zwiers. Uitgave Mij. voor goede en goedkoope Lectuur. 203 pp. F. 3.75. For review see *Chem. Weekblad* 17, 523(1920).

SMYTHE, J. A.: *Lead: Including Lead Pigments and the Desilverization of Lead*. London: Sir Isaac Pitman & Sons., Ltd. 120 pp. 3s. net. For review see *Nature* 106, 241(1920).

Ore-concentrating apparatus. J. E. ARMITSTEAD. U. S. 1,356,332, Oct. 19.

Ore-concentrating apparatus. B. H. DOSENBACH. U. S. 1,354,031, Sept. 28.

Briqueting ores. W. TYRRILL. U. S. 1,356,100, Oct. 19. See Can. 195,569 (C. A. 14, 528).

Platinum from ores. R. THAYER. U. S. 1,355,186, Oct. 12. Pt and similar metals are volatilized from their ores in the form of a fume by treating the ore with  $\text{CaCl}_2$ ,  $\text{NaCl}$  or  $\text{CaF}_2$  and steam or  $\text{H}_2\text{O}$  and then heating it, the fume is entrained in an absorption liquid such as  $\text{H}_2\text{O}$  and metal values are recovered from the enriched liquid electrolytically.

Apparatus for recovering gold by amalgamation. F. M. LEWIS. U. S. 1,354,497, Oct. 5.

Elongated reduction furnace for smelting lead ores. J. LABARTHE. U. S. 1,356,159, Oct. 19.

Separating ores. H. SCHRANZ. Brit. 148,168, July 9, 1920. A carrier is formed of material, such as stone, which is porous to liquids or gas but does not allow the solid particles to pass through. A suitable construction is specified.

Treating silver ores. L. P. BURROWS. U. S. 1,355,795, Oct. 12. Steam is suddenly expanded, sepd. without condensation from any water particles held in suspension, highly heated and brought into contact with crushed Ag ore at a red heat. The ore is smelted and the products are allowed to cool; this results in their sepg. into different layers, containing sepd. metals.

Casting metals. H. E. PAYNE. U. S. 1,355,255, Oct. 12. In casting Fe, stellite or similar metals around a core of Fe, steel, worn tools, etc., the core is coated with graphite, then heated to a cherry-red, placed in a mold and the metal cast around it. This serves to avoid fracture of the cast metal due to contraction on cooling.

**Refining iron.** A. JACKSON. Brit. 147,999, Apr. 25, 1919. Refined Fe obtained by melting pig-iron and steel scrap in a cupola has the following percentage compn.: C 1.5–2.5, Si 0.3–0.6, P 0.3–0.6, and S below 0.1. The Fe is suitable for use in dry and wet puddling. The pig used may contain 1–1.6% each of Si and P.

**Iron and steel bars from scrap metal.** L. JONES. U. S. 1,354,492, Oct. 5. Scrap metal of high and low C content is mixed, the scrap is compressed while cold under sufficient pressure to form slugs or billets without apparent visible interstices, and the slugs or billets are then heated to substantially a welding temp., and rolled into bars.

**Applying a white metal coating to steel or iron surfaces.** K. HAAS. Ger. 302,440, Dec. 2, 1914. The articles, before cleansing, are subjected to the action of strongly reducing or oxidizing fluxes, and are then immediately brought into contact with the molten white metal, with exclusion of air. Boric or phosphoric acids or their salts are specified as suitable reducing or oxidizing agents. In tinning,  $\text{SnCl}_2$ , to which  $\text{ZnCl}_2$  and  $\text{NH}_4\text{Cl}$  may be added, is used. Pb is applied with the use of  $\text{PbCl}_2$  (if necessary with the addition of  $\text{ZnCl}_2$  and  $\text{NH}_4\text{Cl}$ ) as flux.

**Nickel-steel.** P. F. COWING. U. S. 1,356,342, Oct. 19. Ni steel is produced by electrolytically dissolving a Ni-Cu alloy obtained from Ni-Cu mat to form a soln. containing Ni and Cu, substituting Fe (or Ni and Fe) for the Cu in the soln., electrolytically depositing Ni-Fe from the soln. and incorporating it with steel.

**Iron castings.** A. DIEFENTHALER. Brit. 147,933, July 9, 1920. Gray-Fe castings for machine parts required to withstand friction are formed of comparatively pure metal, and are cooled in the mold so as to obtain a pearlite structure with exclusion of ferrite.

**Casting aluminium and its alloys.** METALLINDUSTRIE SCHIELE & BRUCHSALER. Brit. 147,723, July 8, 1920. Addition to 137,325 (C. A. 14, 1296). The invention described in the principal patent is applied to sand or clay molds and cores which have been dried slowly at 400°.

**Aluminium alloys.** METALLBANK UND METALLURGISCHE GES. Brit. 147,903, July 9, 1920. Al is alloyed with Li, which may be partly or wholly replaced by Ba or Sr with or without the addition of other metals such as Cu, Zn, Mg, Sn, etc. The alloys may contain up to 40% of Li, and may be annealed one or more times by heating to over 100° and cooling in air or liquid, or otherwise.

**Iron alloy.** G. H. CHARLES. U. S. 1,355,589, Oct. 12. A rust-resisting alloy is formed of Fe combined with about 0.15% of Mo and not over 0.20% of C. U. S. 1,355,590 relates to the formation of a similar alloy by the addition of about 0.15% of Mo to molten Fe. The alloy thus formed is suitable for rolling into sheets.

**Iron or steel alloy.** K. IBE. U. S. 1,356,367, Oct. 19. See Brit. 131,492 (C. A. 14, 270).

**Alloys of nickel.** P. H. BRACE. U. S. 1,355,532, Oct. 12. In forming alloys of Ni with Ta and Cb, the oxides of the metals to be alloyed are mixed, reduced with an excess of C to form carbides and the latter are subsequently melted *in vacuo* in the presence of Ta oxide. A silvery white malleable alloy is thus obtained.

**Alloy of gold and nickel.** F. E. CARTER. U. S. 1,355,811, Oct. 19. An alloy formed of Au 83.3 and Ni 16.7% has a color resembling Pt and is capable of being worked substantially in the same manner as Pt. It is suitable for use in the manuf. of jewelry.

**Copper-nickel alloy.** F. MILLIKEN. U. S. 1,354,988, Oct. 5. An alloy adapted for resisting some hot acids is formed of Cu 50–60, Ni 28–36, Zn 4–8 and Fe 4–8%.

**Lead-copper-nickel-zinc alloy.** F. MILLIKEN. U. S. 1,354,989, Oct. 5. An alloy suitable for valve fittings is formed of Pb 10–14, Cu 55–65, Ni 6–11 and Zn 14–18%.

**Copper-nickel-chromium-iron alloy.** F. MILLIKEN. U. S. 1,354,990, Oct. 5,

An alloy adapted for resisting hot organic acid solns. is formed of Fe 16-20, Cr 5-7, Cu 31-38, Ni 38-46 and Mn 0.25-0.75%.

**Copper alloy.** A. G. MUMFORD. U. S. 1,355,769, Oct. 12. An alloy suitable for cast feed water regulator floats is formed of Cu 80.36% and 19.64% of a preformed white metal alloy composed of Sn 61, Cu 29.7 and Zn 9.3 parts.

**Ferromanganese.** J. E. JOHNSON, JR. U. S., 1,354,490, Oct. 5. Alloys of Fe high in Mn are produced in a blast furnace by blowing the furnace charge containing Fe, Mn and C with a blast richer in O and poorer in N than air and so proportioning the O and N as to effect an increase in hearth temp. sufficient to obtain the desired high Mn content in the alloy. The amt. and temp. of the gases supplied are occasionally decreased to diminish Mn losses and effect a saving of fuel.

**Ferrochromium.** J. E. JOHNSON, JR. U. S. 1,354,491, Oct. 5. A charge containing Fe, Cr and C is blown in a blast furnace with a blast richer in O and poorer in N than air, regulated to produce a sufficient increase in hearth temp. to obtain the desired high Cr content in the alloy.

**Alloy for soldering internal-combustion-engine cylinders.** M. A. WILLOUGHBY. U. S. 1,355,202, Oct. 12. An alloy adapted for repairing scratches, channels or holes in internal-combustion-engine cylinders is formed of Sn 79.15, Sb 7.29, Cu 8.49 and Zn 7.07 parts.

**Apparatus for testing hardness of metals.** E. SCHNEIDER. U. S. 1,354,216-7-8, Sept. 28.

**Preventing oxidation or rusting of metal.** N. C. F. JENSEN. U. S. 1,354,487, Oct. 5. Oxidation is prevented by immersing the metal in a bath containing ordinary lubricating oil, draining off superfluous oil, heating the treated metal to about 300° until it assumes a grayish or ash color and then, without previous cooling, immersing it in an oily bath and draining and cooling.

**Welding electrode.** G. W. PETERS and O. F. ALKIRE. U. S. 1,356,468, Oct. 19. Electrodes for welding are formed of a tube of welding material such as cold-rolled metal with an inner coating of flux.

**Electrode for arc-welding.** R. C. KNOLL, F. J. RODE and C. KOTCHL. U. S. 1,354,664, Oct. 5. A welding electrode is formed of a rod of metal having annular grooves and a covering of wood pulp or other protective material.

**Electrode for arc welding.** J. W. FAY. U. S. 1,354,476, Oct. 5. An electrode suitable for welding is formed of a bar of metal surrounded by successive coatings of cement, paper and paste, which serve to maintain a uniform action by the electrode as it is consumed.

**Electrically welding steel.** J. W. PLANT. U. S. 1,354,266, Sept. 28. The welding is carried out in a chamber from which oxidizing gases are excluded, to obviate slag formation such as might weaken the weld.

## 10—ORGANIC CHEMISTRY

C. A. ROULLER

**Emil Fischer memorial lecture.** MARTIN O. FORSTHER. *J. Chem. Soc.* 117, 1157-1201(1920).—Following a biographical sketch is given a thorough survey of Fischer's invaluable work in org. and biological chemistry. E. J. C.

**Structure symbols of organic compounds and radicals.** INGO W. D. HACKH. *Chem. Age (London)* 3, 392-4(1920).—A system of notation is described in which each compd. and radical has a symbol that indicates its structure. E. H.

**Catalytic hydrolysis of nitriles.** A. MAHLE. *Bull. soc. chim.* 27, 754-6(1920).—  
See C. A. 14, 3227. E. H.

**Ethylene sulfide.** MARCEL DELAFINE. *Bull. soc. chim.* 27, 740-3 (1920).—See C. A. 14, 3226. E. H.

**Ether apparatus and stray electric currents.** OSCAR SCHAAAL. *Chem.-Ztg.* 44, 741 (1920).—The rapid destruction of lead stills by electrolysis is affirmed. Exptl. proof is adduced to show that in the presence of the acid liquor currents are generated where (a) hard and soft lead are joined, and (b) a carbon-bearing resinous deposit forms on the lead. It is supposed that thermoelec. action may be responsible for a part of the corrosion. W. C. EBAUGH

**Cyanoacetylene.** CHARLES MOUREU AND JACQUES CH. BONGRAND. *Ann. chim.* 14, 47-58 (1920).—Ten g. HC:CCO<sub>2</sub>H (A) (prepd. by the interaction of alc. KOH and (CHBrCO<sub>2</sub>H)<sub>2</sub> according to the method of Perkin and Simonson (C. A. 1, 2684)) in dry Et<sub>2</sub>O poured into 4% NH<sub>3</sub> in abs. EtOH, gave 8.6 g. HC:CCO<sub>2</sub>NH<sub>4</sub>, white needles from abs. EtOH, which on dehydration would not give HC:C CONH<sub>2</sub> (B) or HC:CCN (C). A with abs. EtOH or MeOH, in the presence of H<sub>2</sub>SO<sub>4</sub>, gave HC:CCO<sub>2</sub>Et, b<sub>760</sub> 119-20°, or HC:CCO<sub>2</sub>Me, b<sub>755</sub> 101.5-2°. On treating these esters with NH<sub>4</sub>OH cooled to -5°, propiolamide (B) was formed, white plates, m. 61-2°, very sol. in H<sub>2</sub>O, EtOH and Et<sub>2</sub>O, slightly sol. in CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub> and CS<sub>2</sub>. B with NH<sub>4</sub>-CuCl<sub>2</sub> formed a yellow ppt., which, on heating, easily caught fire, and probably consisted of a mixt. of the Cu salts, (CuC:CCONH<sub>2</sub>)<sub>2</sub>, and HOCuC:C CONH<sub>2</sub>. B with aq. AgNO<sub>3</sub> gave AgC:C CONH<sub>2</sub>, with NH<sub>4</sub>-AgNO<sub>3</sub> a mixt. of AgC:C CONH<sub>2</sub> and AgC:C CONH<sub>2</sub> + AgOH, and with alc. AgNO<sub>3</sub>, AgC:C CONH<sub>2</sub> + AgNO<sub>3</sub> containing some AgC:C CONH<sub>2</sub>. These Ag compds., which formed as white ppts., caught fire on moderate heating. The dehydration of B with P<sub>2</sub>O<sub>5</sub> by the method and app. used in the prepn. of C<sub>2</sub>N<sub>2</sub> (*Ann. chim.* 14, 5-46) gave white crystals of cyanoacetylene (C), b<sub>760</sub> 42.5°, m. 5°, d<sub>4</sub><sup>17</sup> 0.8159, n<sub>D</sub><sup>17</sup> 1.38399, n<sub>B</sub><sup>17</sup> 1.38699, n<sub>F</sub><sup>17</sup> 1.39440, n<sub>A</sub><sup>17</sup> 1.40072, slightly sol. in H<sub>2</sub>O, easily sol. in alc. The liquid and its solns. rapidly turned brown. It irritated the mucous membranes, caused lachrymation, and was easily inflammable, producing a smoky flame in insufficient air and a purple flame resembling that of C<sub>2</sub>N<sub>2</sub> and C<sub>2</sub>N<sub>2</sub> in excess of air. KOH attacked it readily, forming a tarry mass. With ammoniacal, alc. or aq. AgNO<sub>3</sub> it gave a white ppt. which exploded easily and contained 61-68% Ag. Ammoniacal Cu salts caused the formation of the Cu compound HOCuC:C CN, an easily inflammable olive-green substance, which with K<sub>3</sub>Fe(CN)<sub>6</sub> gave a small amt. of very volatile and unstable white crystals, m. 64°, with an odor and irritating properties resembling C<sub>2</sub>N<sub>2</sub> and C<sub>2</sub>N<sub>2</sub>, and was probably another subnitride, C<sub>2</sub>N<sub>2</sub>. G. W. STRATTON

**A new way of forming hexamethylenetetramine.** TH. SABALITSCHKA. *Z. angew. Chem.* 33, I, 217 (1920).—The formation of (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub> by the interaction of (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> and HCHO (C. A. 14, 2174) and by the interaction of NH<sub>4</sub> salts and formalin has been observed (*Z. angew. Chem.* 33, I, 84 (1920); C. A. 2, 1944). In order to det. more definitely whether formalin will act upon NH<sub>4</sub> salts directly, S. added to solns. of formalin neutralized with alkali and containing Me orange neutral solns. of NH<sub>4</sub>Cl and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. In each case the soln. turned red, indicating that the reaction was taking place. G. W. STRATTON

**The use of catalysts in the sulfonation of aromatic compounds.** JOS. A. AMBLER AND WM. J. CORTON. *J. Ind. Eng. Chem.* 12, 968-9 (1920).—The accelerating effect of CuSO<sub>4</sub>, HgSO<sub>4</sub>, V<sub>2</sub>O<sub>5</sub>, Na<sub>2</sub>SO<sub>4</sub>, CrO<sub>3</sub>, K<sub>2</sub>SO<sub>4</sub>, Li<sub>2</sub>SO<sub>4</sub> and mixts. of Na<sub>2</sub>SO<sub>4</sub> and V<sub>2</sub>O<sub>5</sub> on the sulfonation of C<sub>6</sub>H<sub>6</sub> was studied by the continuous process of Ambler and Gibbs (U. S. Pats. 1,292,950, 1,300,227 and 1,300,228). In each expt. the catalyst was dissolved in 70% H<sub>2</sub>SO<sub>4</sub> and the sulfonation carried on for 1 to 1.5 hrs. at temps. of 242° to 260°. The sulfonation was measured by finding the amt. of C<sub>6</sub>H<sub>5</sub> sulfonated per min. and the amts. of mono- and di-SO<sub>3</sub>H acids formed per min. All of the substances used increased the amt. of C<sub>6</sub>H<sub>5</sub> sulfonated per min., the most active catalyst being a mixt. of Na<sub>2</sub>SO<sub>4</sub> and V<sub>2</sub>O<sub>5</sub> in any reasonable proportions. Na<sub>2</sub>SO<sub>4</sub> and K<sub>2</sub>SO<sub>4</sub>

had a distinct catalytic effect not entirely due to the increased b. p. of the  $\text{H}_2\text{SO}_4$ . The amt. of  $\text{di-SO}_3\text{H}$  acid formed per min. was increased by  $\text{Na}_2\text{SO}_4$  and  $\text{Li}_2\text{SO}_4$  and a mixt. of  $\text{Na}_2\text{SO}_4$  and  $\text{V}_2\text{O}_5$  was still more effective. The other catalysts used inhibited this reaction. A short historical review of catalytic sulfonation, with references, is given.

G. W. STRATTON

The solubility of  $\beta, \beta'$ -dichloroethyl sulfide in petroleum hydrocarbons and its purification by extraction with these solvents. THOS. G. THOMPSON AND HENRY ODEHN. *J. Ind. Eng. Chem.* 12, 1057-62(1920).—Soly. expts. gave the following values for the critical temps. of soly. of mustard gas in these hydrocarbons: ligroin,  $19^\circ$ ; gasoline,  $20.4^\circ$ ; kerosene,  $25.6^\circ$ ; railroad light oil,  $37^\circ$ . In these solvents  $\text{S}_2\text{Cl}_2$  was found to be sol. in all proportions above  $0^\circ$ , while S was only slightly sol. In railroad light oil chlorinated mustard gas was sol. in all proportions above  $8.8^\circ$ . Mustard gas was extd. from the crude material by using ligroin as a solvent in different lab. app. for single, successive or continuous extn. The extn. was also carried out on a semi-con. scale in a continuous extn. app., using railroad light oil as a solvent. It was found possible to obtain a 54 to 69% yield of mustard gas from crude material containing 71%.

G. W. STRATTON

Pressures produced by the action of sulfur monochloride on  $\beta, \beta'$ -dichloroethyl sulfide. THOS. G. THOMPSON AND F. J. KOPP. *J. Ind. Eng. Chem.* 12, 1056-7(1920).—The pressures produced by the gases formed in the interaction of varying concns. of  $\text{S}_2\text{Cl}_2$  and  $(\text{C}_2\text{H}_4\text{Cl})_2\text{S}$  were measured by manometer tubes attached to the reaction flasks. When rather large amts. of the mustard gas and small amts. of  $\text{S}_2\text{Cl}_2$  were allowed to interact the max. pressures were reached at the end of three days, after which there was a decrease in pressure. This indicated a decided interaction with a secondary action occurring after three days.  $\frac{1}{2}\text{H}_2\text{S}$  and  $\text{HCl}$  were detected in the gases formed.

G. W. STRATTON

Decomposition of and pressure developed by mustard gas in steel shell at  $60^\circ\text{C}$ . W. A. FELSING, H. ODEHN AND C. B. PETERSON. *J. Ind. Eng. Chem.* 12, 1063-5 (1920).—In one series of expts. 300-cc. samples of  $(\text{C}_2\text{H}_4\text{Cl})_2\text{S}$  were placed in 75 mm. steel shells which were closed by rubber stoppers and held at  $60^\circ$  for 16 days. Samples of the liquid were removed every two days and tested for m. p. and acidity. In the other series of expts. the shells containing the  $(\text{C}_2\text{H}_4\text{Cl})_2\text{S}$  were connected with Hg manometers and held at  $60^\circ$ , the pressures developed being measured twice daily. The results which are given in tables and curves showed that the acidity (due to  $\text{HCl}$ ) increased with time, but did not become great enough to cause serious corrosion. The m. p. was lowered as the acidity increased. The pressures developed, which reached a max. after about 9 days, did not exceed 2 atm. S was deposited in amts. of 5 to 8% of the liquid contents of the shells. A preliminary treatment with  $\text{NH}_3$  delayed the formation of pressure.

G. W. STRATTON

The precipitation of sulfur from crude mustard gas by means of ammonia. W. A. FELSING AND S. B. ARENSEN. *J. Ind. Eng. Chem.* 12, 1065-6(1920).—The equation for the reaction of  $\text{S}_2\text{Cl}_2$  and  $\text{C}_2\text{H}_4$  to form  $(\text{C}_2\text{H}_4\text{Cl})_2\text{S}$  shows that 16.76% of the mixt. is free S. In one process (60° process) of manuf. 2 to 8% of this S seps. out and an additional amt. crysts. when the product is subjected to moisture, freezing or long standing. Moist  $\text{NH}_3$  causes the pptn. of S to the extent of 5 to 10% of the mixt., while with dry  $\text{NH}_3$  and dry  $(\text{C}_2\text{H}_4\text{Cl})_2\text{S}$  no pptn. occurs. In the other process (30 to  $35^\circ$  or Levinstein process) of manuf. no S seps., and when the product is subjected to moisture, freezing or long standing the S pptn. is only slight. Moist  $\text{NH}_3$  causes the pptn. of 40 to 45% of the supposedly free S in this product, the m. p. of which is about the same before and after pptn. This and other facts indicate that only 40 to 45% of the "free" S is present in colloidal form, the remainder being in compds.

G. W. STRATTON

**The intersolubility of chloropicrin and water.** THOS. G. THOMPSON AND JOHN H. BLACK. *J. Ind. Eng. Chem.* 12, 1066-7(1920).—The amt. of  $\text{CCl}_3\text{NO}_2$ , dissolved in  $\text{H}_2\text{O}$  was detd. by treating the soln. with alc.  $\text{Na}_2\text{SO}_3$ , refluxing, evapg. the  $\text{C}_2\text{H}_5\text{OH}$ , concg. the soln., adding  $\text{AgNO}_3$  in excess to ppt. the Cl and titrating the excess of  $\text{AgNO}_3$  with  $\text{NH}_4\text{CNS}$ . The soly. was only slight, decreasing with increase in temp. The soly. of  $\text{H}_2\text{O}$  in  $\text{CCl}_3\text{NO}_2$  was detd. by placing small amts. of  $\text{H}_2\text{O}$  in large amts. of  $\text{CCl}_3\text{NO}_2$ , heating the mixt. until dissoln. occurred, then cooling until  $\text{H}_2\text{O}$  sepd., taking the mean of the temps. noted as the temp. of miscibility. The soly. was slight, increasing with increase in temp. G. W. STRATTON

**Note on catalysis in the manufacture of ether.** HUGO SCHLATTER. Hercules Powder Co. *J. Ind. Eng. Chem.* 12, 1101-2(1920).—The statement (cf. C. A. 4, 3205) that  $\text{Al}_2(\text{SO}_4)_3$  in the usual  $\text{H}_2\text{SO}_4$ -EtOH mixt., due to a double compd. formation lowers the temp. at which regular and rapid evolution of  $\text{Et}_2\text{O}$  takes place is shown to be erroneous; the salt has in addition a harmful effect upon the Pb coils in the still by causing severe pitting. The lowering of temp. of reaction is due not to a double compd., but to a physical or surface action, as broken porcelain has the same effect. The formation of  $\text{PbSO}_4$  which normally occurs in the ordinary method of manuf. has this same effect. N. A. LANGE

**Control experiments in chloropicrin manufacture. I. The effect of varying quantities of lime upon the yield of chloropicrin.** H. L. TRUMBULL, G. T. SOHL, W. I. BURT AND S. G. SEATON. Edgewood Arsenal. *J. Ind. Eng. Chem.* 12, 1068(1920).—Expts. closely approximating actual manufg. practice showed that an excess of lime had no influence upon the yield of chloropicrin; the hydrolysis in aq. soln. is the same whether  $\text{Ca}(\text{OH})_2$  be present or not present. The yield is favorably influenced when the mixt. is cooled below  $30^\circ$  during the picric acid or picrate addition to the  $\text{CaOCl}_2$  soln. **II. Active chlorine in the sludge from the manufacture of chloropicrin.** H. L. TRUMBULL, S. G. SEATON AND HOWARD DURHAM. *Ibid* 1068-9.—Expts. were made to det. the approx. rate at which active Cl disappears during steam distn. With an initial concn. of 6.98 and 6.18% active Cl, after 1 hr. steam distn. 2.52 and 3.21% and after 2 hrs. 0.714 and 0.747% active Cl remained, the rate of decompn. increasing with higher initial concns. Samples of the sludge left in the chloropicrin stills at the end of a distn. showed 0.28-0.32% active Cl. N. A. LANGE

**Naphthalenesulfonic acid. I. Some difficultly soluble salts of certain naphthalene-sulfonic acids.** JOSEPH A. AMBLER. U. S. Bur. Chem. *J. Ind. Eng. Chem.* 12, 1080-5(1920).—The purpose of the investigation was to obtain a characteristic salt of each acid which might serve as a qual. test. The salts were prepd. by mixing hot aq. solns. of mol. equiv. quantities of the acid or its Na or K salts and the HCl salt of the organic base; the salts were filtered, washed with cold  $\text{H}_2\text{O}$  and dried *in vacuo* at  $100^\circ$ . Since they are salts of strong acids and weak bases, a small amt. of the HCl salt of the latter is added when recrystg. The salts mentioned below are described in detail with crystallographic properties, refractive indices and the action with polarized light.  $\alpha$ -Naphthylamine naphthalene- $\alpha$ -sulfonate m.  $232^\circ$  (decompn.) and is sol. in hot  $\text{H}_2\text{O}$ , hot EtOH, insol. in cold  $\text{H}_2\text{O}$ , EtOH or  $\text{Me}_2\text{CO}$ .  $\beta$ -Naphthylamine salt begins to turn brown at  $202^\circ$  and m.  $211^\circ$ ; it is sol. in hot  $\text{H}_2\text{O}$ , hot  $\text{Me}_2\text{CO}$  or cold EtOH- $\text{H}_2\text{O}$  (4:1) and slightly sol. in cold EtOH,  $\text{H}_2\text{O}$  or  $\text{Me}_2\text{CO}$ .  $\alpha$ -Naphthylamine naphthalene- $\beta$ -sulfonate m.  $240-2^\circ$  (decompn.); it is sol. in cold EtOH- $\text{H}_2\text{O}$  (4:1), fairly sol. in hot  $\text{H}_2\text{O}$  or EtOH, slightly sol. in cold  $\text{H}_2\text{O}$ , EtOH or  $\text{Me}_2\text{CO}$ .  $\beta$ -Naphthylamine salt m.  $276-9^\circ$  (decompn.); it is readily sol. in hot EtOH- $\text{H}_2\text{O}$  (4:1), slightly sol. in hot  $\text{H}_2\text{O}$  or EtOH and difficultly sol. in cold  $\text{H}_2\text{O}$  or  $\text{Me}_2\text{CO}$ . Ferrous salt,  $\text{Fe}(\text{C}_{10}\text{H}_7\text{SO}_3)_2 \cdot 6\text{H}_2\text{O}$  (cf. Brit. Pat. 4459(1894)), is a remarkably stable salt which loses  $\text{H}_2\text{O}$  at  $150-60^\circ$ , readily sol. in hot and slightly sol. in cold  $\text{H}_2\text{O}$ .  $\alpha$ -Naphthylamine naphthalene-1,5-disulfonate does not m. below  $280^\circ$ ; it is very slightly sol. in hot  $\text{H}_2\text{O}$ , EtOH or



EtOH-H<sub>2</sub>O (4:1) and insol. in these cold solvents and in Me<sub>2</sub>CO. *β*-Naphthylamine salt m. above 280° and shows solubilities similar to the preceding salt. *α*-Naphthylamine naphthalene-1,6-disulfonate m. 265-7° (decomp.); it is sol. in hot H<sub>2</sub>O, EtOH and in cold EtOH-H<sub>2</sub>O (4:1), slightly sol. in cold EtOH and insol. in cold H<sub>2</sub>O or Me<sub>2</sub>CO. *β*-Naphthylamine salt, m. above 280°; it is sol. in hot H<sub>2</sub>O or EtOH, slightly sol. in cold EtOH and insol. in cold H<sub>2</sub>O or Me<sub>2</sub>CO; sol. in cold EtOH-H<sub>2</sub>O (4:1). *α*-Naphthylamine naphthalene-2,6-disulfonate m. above 280°; it is fairly sol. in hot H<sub>2</sub>O, slightly sol. in hot EtOH and insol. in cold H<sub>2</sub>O, EtOH or Me<sub>2</sub>CO; sol. in hot EtOH-H<sub>2</sub>O (4:1). *β*-Naphthylamine salt m. above 280°; it is but slightly sol. in hot and insol. in cold solvents. *α*-Naphthylamine naphthalene-2,7-disulfonate decomps. gradually without m. above 220°; it is sol. in hot H<sub>2</sub>O, in EtOH or in cold EtOH-H<sub>2</sub>O (4:1) and insol. in cold H<sub>2</sub>O or Me<sub>2</sub>CO. *β*-Naphthylamine salt m. above 280°; it is sol. in hot and insol. in cold H<sub>2</sub>O or EtOH, insol. in Me<sub>2</sub>CO and fairly sol. in cold EtOH-H<sub>2</sub>O (4:1). II. A method for the qualitative detection of some of the naphthalenesulfonic acids. JOSEPH A. AMBLER AND EDGAR T. WHERRY. *Ibid* 1085-7.—FeCl<sub>3</sub> is added to the cold soln., pptg. Fe<sup>3+</sup> naphthalene-β-sulfonate, filtered and the filtrate concd. to the original vol. A portion of the filtrate is tested for 1,5-C<sub>10</sub>H<sub>6</sub>(SO<sub>3</sub>H)<sub>2</sub> by pptg. with α-C<sub>10</sub>H<sub>7</sub>NH<sub>2</sub>.HCl from a hot soln.; if present it is completely removed from the remainder; the excess of the base is removed by titration with NaOH, and phenolphthalein and filtering the free base pptd.; after acidification with HCl, the soln. is heated to boiling and an excess of β-C<sub>10</sub>H<sub>7</sub>NH<sub>2</sub>.HCl is added to form salts with all SO<sub>3</sub>H acids remaining; the 2,6-acid is indicated by a white ppt. formed while boiling; after filtering hot it is allowed to cool and any ppt. which forms is dried and then extd. with hot Me<sub>2</sub>CO to remove the α-SO<sub>3</sub>H acid; the salts insol. in Me<sub>2</sub>CO are dried and examd. under the microscope.

N. A. LANGE

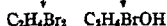
**Action of chlorine on 3,5-dichloro-1,1-dimethyl-Δ<sup>3,4</sup>-cyclohexadiene.** LEONARD E. HINXEL. *J. Chem. Soc.* 117, 1296-1303 (1920).—The action of Br (1 and 2 mols.) on 3,5-dichloro-1,1-dimethyl-Δ<sup>3,4</sup>-cyclohexadiene (A) has been investigated by Crossley (*J. Chem. Soc.* 85, 264 (1904)) and the action of Cl on A was studied to throw more light on the mechanism of the reaction. When A in dry CHCl<sub>3</sub> is treated with Cl, HCl is evolved and after removal of the solvent the yellow product deposited *in vacuo* over NaOH in colorless prisms, m. 103.5°, is 2,3,3,4,5-pentachloro-1,1-dimethyl-Δ<sup>3,4</sup>-cyclohexene (B), sol. in Et<sub>2</sub>O, CHCl<sub>3</sub>, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. When B is heated at 120-30° HCl is evolved and on cooling crystals of 1,2,3,4,5-C<sub>6</sub>HMe<sub>2</sub>Cl<sub>5</sub> (C) sepd. (*Ber.* 18, 1369 (1885)). B interacted vigorously with fuming HNO<sub>3</sub> and the product treated with H<sub>2</sub>O, washed and crystd. from AcOEt, m. 223-4°, and is 3,4,5,6-Cl<sub>4</sub>C<sub>6</sub>Me<sub>2</sub> (D). The mother liquors from which B sepd. gave off HCl when heated to 180-200°, and when distd. gave 3,5,6-trichloro-o-xylene (E), m. 47.5°, and also C and D. When C was treated in dry CHCl<sub>3</sub> with Fe and Cl, D resulted and with Br gave needles, m. 226°, of 3,4,5-trichloro-6-bromo-o-xylene (F), readily sol. in Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, and sparingly sol. in CHCl<sub>3</sub> and alc. When C is treated with fuming HNO<sub>3</sub> and the product is washed with H<sub>2</sub>O and crystd. from alc. stout yellowish needles, m. 149°, of 3,4,5-trichloro-6-nitro-o-xylene (G) are formed. This product is sol. in Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub> and light petroleum. C was prepd. from o-4-xylidene (Crossley, *loc. cit.*) by acetylating, chlorinating and treating in HCl with CuCl and NaNO<sub>2</sub>. When A in cold CHCl<sub>3</sub> was treated with Cl, no cryst. product could be secured and on distg. the liquid, 3 fractions (1) 217-20°, (2) 222-6°, and (3) 228-33°, were obtained. Fraction 2 gave a product which on treatment with Br and Fe gave 3,5,4,6-Cl<sub>2</sub>Br<sub>2</sub>C<sub>6</sub>Me<sub>2</sub> (H) (*loc. cit.*) and fraction 3 contained E. When E was treated in CHCl<sub>3</sub> with Br and Fe, H was formed, and when treated with fuming HNO<sub>3</sub> the resulting product was 3,5,4,6-Cl<sub>2</sub>(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>Me<sub>2</sub>. H. E. WILLIAMS

**Some new azopyrazolones and allied compounds.** KENNETH H. SAUNDERS. *J. Chem. Soc.* 117, 1284-72 (1920).—In seeking to study, by a comparison of the ab-

sorption spectra, the effect of increased mol. wt. on the color of the compds. of the azo type belonging to the  $C_6H_4$ ,  $C_{10}H_8$ , and anthraquinone groups, a scarcity of compds. was experienced. Although azobenzene and azonaphthalenes are well known, azo-anthraquinone is comparatively new (Gattermann and Ebert, *C. A.* 17), and the azoanthracenes are unknown. Azopyrazolones, since they are strongly colored and faster to light than the corresponding members of the azobenzene series, seemed good prospects for investigation. A complete series of compds. was prepd. in which the anthraquinone nucleus is introduced either into the 1-position of the pyrazolone ring, or as the first component of an azopyrazolone compd., or in both positions together. In making large amts. of anthraquinonylhydrazine (A) the synthesis by Mohlau, Viertel and Reiner (*C. A.* 6, 2930, 2931) from chloroanthraquinone could not be used owing to the low (20%) yields. Anthraquinonediazonium sulfate may be reduced in aq.  $KHSO_4$ , followed by hydrolysis of the hydrazinedisulfonic acid by HCl. The process is lengthy, since the sulfate is sparingly sol. Aminoanthraquinone (B) in excess of HCl can be diazotized at ordinary temp. as readily as in  $H_2SO_4$ .  $SnCl_4$  gives poor yields of the hydrazine, due probably to a 2-fold cause. The anthraquinone C:O group is more easily reduced than the diazo group and steric hindrance causes general sluggishness of the  $\alpha$ -H in the hydrazine, so that fission of the diazonium chloride results.  $SnCl_4$  (1 mol.) was added and it caused a smoother reaction. A was condensed with  $AcCH_2CO_2Et$  in  $AmOH$  and the hydrazone after recrystn. from alc. was treated with  $Ac_2O$ . The acetylpyrazolone was hydrolyzed by boiling it 2 hrs. with HCl and  $AcOH$  and 1-anthraquinonyl-3-methyl-4-pyrazolone (C) resulted, yellow-red gelatinous mass, sol. in glacial  $AcOH$ , yielding an orange soln. and red crystals, insol. in  $H_2O$ . A red soln. resulted with  $H_2SO_4$ . In  $AcOH$  C gave with  $HNO_3$  a pale yellow isonitroso compound. When C in glacial  $AcOH$  is treated with  $PhN_2Cl$  a yellow gelatinous mass seps. This, when crystd. from  $C_6H_5N$ , gave yellow needles of 4-benzeneazo-1-anthraquinonyl-3-methyl-5-pyrazolone (D). 4- $\alpha$ -Naphthaleneazo-1-anthraquinonyl-3-methyl-5-pyrazolone and the corresponding  $\beta$ -compound were prepd. in the same way.  $\beta$ -Aminoanthraquinone-HCl was diazotized in glacial  $AcOH$  with excess  $AmNO_2$  (*Z. Farben. Ind.* 2, 469(1903)) and combined with C, giving 4- $\beta$ -anthraquinoneazo-1- $\beta$ -anthraquinonyl-3-methyl-5-pyrazolone, yellow needles from  $PhNO_2$ , but it was insol. in all other common solvents. B as the sulfate was diazotized, yielding the yellow stable cryst. diazo compd. (87% yield) and this was treated in  $NaOAc$  soln. with 3-methyl-5-pyrazolone. Yellow flakes of 4- $\beta$ -anthraquinoneazo-3-methyl-5-pyrazolone (E) were formed. This product is sparingly sol. in  $PhNO_2$  and  $AmOH$ . When 1- $\beta$ -naphthyl-3-methyl-5-pyrazolone in glacial  $AcOH$  interacted with B as the diazonium compd., 4- $\beta$ -anthraquinoneazo-1- $\beta$ -naphthyl-3-methyl-5-pyrazolone (F) sepd. as a flocculent orange mass, brick-red needles from  $PhNO_2$ . B as the diazonium sulfate,  $NaOAc$  and  $AcCH_2CO_2Et$  gave ethyl  $\beta$ -anthraquinoneazoacetate (G), yellow powder, m. 216–8°, sol. in alc.,  $CHCl_3$ ,  $C_6H_6$ ,  $C_7H_8$ ,  $PhNO_2$  and  $C_6H_5N$ . G in glacial  $AcOH$  and aq.  $NaOAc$  with aq.  $NH_4OH$ . HCl deposited orange needles, m. 188–91°, of 4- $\beta$ -anthraquinoneazo-3-methyl isoxazolone. With  $N_2H_4$  E was formed.

H. E. WILLIAMS

Preparation and characterization of ethylenbromohydrin. JOHN READ and REXFORD G. HOOK. *J. Chem. Soc.* 117, 1214–26(1920).—Due to the nature of the methods of prepg. ethylenbromohydrin (A) it has hitherto been prepd. only in an impure condition. In the older methods  $C_2H_4(OH)_2$  was the starting material, the second reagent being  $C_2H_4Br_2$  (*Ann. chim. phys.* [3] 67, 275(1863)),  $HBr$  (*Jahresb.* 1872, (304)) or  $PBr_3$  (*Ber.* 9, 48(1876)), and more recently the interaction between  $C_2H_4IOH$  and  $Br$  (*Ber.* 22, 1321(1889)) and the direct action of  $HBrO$  on  $C_2H_4$  (*J. Russ. Phys. Chem. Soc.* 30, 900(1898)). The last method gives the purest product. A study has been made of the reaction between  $C_2H_4$  and dil.  $Br-H_2O$ , the reaction being followed by detg. titrimetrically the  $HBr$  formed, as follows:



Results indicate a remarkable preponderance of A in the initial stages of the reaction. When the concn. of A has reached 0.2 N the mol. ratio of A to  $\text{C}_2\text{H}_4\text{Br}_2$  (B) is about 10:1, and in more dil. soln. the value is still higher. Pure A is prepd. under conditions described previously (C. A. 14, 2481). The liquid which results under these conditions was neutralized by adding solid  $\text{Na}_2\text{CO}_3$  and the aq. layer was sepd. from B. The amt. of HBr produced was detd. by the use of standard  $\text{AgNO}_3$ . A was extd. with  $\text{Et}_2\text{O}$  and after the solvent was evapd. from the dry product ( $\text{Na}_2\text{SO}_4$  used), the crude A was distd. repeatedly under atm. pressure, but the product was impure. The temp. of distn. ranged from 147 to 55° and HBr was evolved during the operation. The purest A obtainable by this method contains both B, HBr and  $\text{H}_2\text{O}$ . Using diminished pressure, the impurities are readily eliminated. After removal of the  $\text{Et}_2\text{O}$ , pure  $\text{Na}_2\text{CO}_3$  is added to correct acidity and a product neutral to litmus is obtained when the material is distd. under reduced pressure. The product,  $b_{11}$  53.5°, gives no opalescence with  $\text{AgNO}_3$ . R. and H. report supplementary work of a quant. nature on the effect of concn. of the reaction products, also of temp. and light. For ascertaining the amts. of A produced, the detn. of HBr by  $\text{AgNO}_3$  proved satisfactory. The mol. ratio of A to B decreases continuously with increasing concn. of A and HBr in the soln. and also with rise of temp. By raising the temp. the reaction takes place more slowly. It has generally been assumed that the addition of  $\text{HClO}$  and  $\text{HBrO}$  to unsatd. compds. progresses more favorably in the dark. Results show that no appreciable decompn. of  $\text{HBrO}$  took place when ice-water, Br and  $\text{C}_2\text{H}_4$  were exposed to direct sunlight. A in  $\text{H}_2\text{O}$  has a sweet burning taste, the vapor of the aq. soln. irritates the eyes and nostrils. A is sol. in most of the common org. solvents. It distils unchanged under 14 mm.;  $d_4^{20}$  1.7902. A refractometric examn. of a series of aq. solns. of A was carried out and the relationship between concn. and refractive index of the solns. has been established. A study of the distn. of aq. soln. of A showed that a const. boiling mixt.,  $b_{762.4}$  99.1°, forms when the concn. of A is 35%. The aq. soln. of A was found to decompose during distn., due to hydrolysis. In the cold hydrolysis is noticeable, though slow, and is hastened greatly by heat, presence of acids and alkalies.

H. E. WILLIAMS

Ring formation. III. Condensation of aromatic amines with  $\alpha$ - and  $\beta$ -diketones and with 4,4'-diacetyldiphenyl. CLARENCE V. FERRISS AND EUSTACE E. TURNER. *J. Chem. Soc.* 117, 1140-51 (1920).—Discrepancies in the work of Cain and Micklethwait (C. A. 8, 3416) on the condensation of benzidine and toluidine with benzil or glyoxal are shown by F. and T. The ketones of the  $\text{Ph}_2$  series were investigated for the purpose of detg. the spatial relation of the 2 Ac groups in  $(4\text{-AcC}_6\text{H}_4)_2$ . This ketone, should Kautler's formula be correct, ought to condense with  $o\text{-C}_6\text{H}_4(\text{NH}_2)_2$ ,

giving a compd. of the constitution  $\begin{array}{c} \text{C}_6\text{H}_4\text{CMe:N} \\ | \\ \text{C}_6\text{H}_4\text{CMe:N} \end{array} \rangle \text{C}_6\text{H}_4$  when a mixt. of the

reagents is warmed in  $\text{AcOH}$ . As in the case of the condensation product of benzil and benzidine, no definite constitution can be ascribed to the product, although evidence points to a compd. formed by condensing 2 mols. of the ketone with 1 of diamine. Benzil was condensed with benzidine as described by C. and M. and benzidine was condensed with glyoxal, but their work could not be duplicated. This was not so when toluidine and  $(\text{CHO})_2$  were condensed.  $\text{CH}_3\text{Ac}$  (B) and  $p$ -toluidine condensed to form 2,4,6-trimethylquinoline. Toluidine (A) in xylene was treated with B and the non-crystalline mass, *monoacetylisopropylidenetoluidine*, was converted into the *p*-nitrobenzylidene derivative by boiling with  $p\text{-NO}_2\text{C}_6\text{H}_4\text{CHO}$ . *N*-*p*-Nitrobenzylidenemonoacetylisopropyl-

*idenetoluidine*, brick-red ppt., m. 245–7°, readily sol. in  $C_6H_6$ , sparingly sol. in alc.  $Me_2CO$ . A and B were heated to boiling for 3 hrs. and the excess of B was distd. off. A yellow cryst. residue, *diacetylisopropylidenetolidine*, m. 108°, was formed. This, with  $H_2SO_4$ , gave 2,4,8,2',4',8'-hexamethyl-6,6'-diquinolyl, cubes from  $C_6H_6$ , m. 252.5°. The *chloroplatinate* was prepd.  $BzCH_2Ac$  and benzidine were heated in  $C_6H_{10}$  and a yellow product formed, *monobenzoylisopropylidenebenzidine*, needles, m. 179°, which with B in  $C_6H_6$  gave *acetylisopropylidenebenzoylisopropylidenebenzidine*, yellow leaflets, m. 234–8°, from  $C_6H_6$ -alc. *Monobenzoylisopropylidenetolidine*, prepd. in a similar way, m. 170°, yellow needles.  $Ph_3$  and  $AcCl$  heated in  $CS_2$  with  $AlCl_3$  gave  $Ac-C_6H_4Ph$  (Adam, *Ann. phys.* [6] 15, 224(1886)) and 4,4'-*diacetyldiphenyl* (D), colorless leaflets, m. 190–1°, sol. in dil.  $AcOH$ , alc. and  $CCl_4$ . The prepn. of D from diazotized 4- $H_2NC_6H_4Ph$  and  $KCN(CN)_2$  proved unsatisfactory. C was prepd. from 4- $NCC_6H_4Ph$ , using  $MgMeI$  and  $Et_2O$ .  $PhC_6H_4COCH_2Ph$  (Papeke, *Ber.* 21, 1339(1888)) was prepd. by condensing  $Ph_2$  with  $PhCH_2COCl$  in  $CS_2$  with  $AlCl_3$  and also by treating 4- $NCC_6H_4Ph$  in  $C_6H_6$  with  $C_6H_5MgCl$  and  $Et_2O$ . (4- $NCC_6H_4$ )<sub>2</sub> (E), hitherto prepd. only from the di- $SO_3H$  acid by fusion with  $KCN$ , may now be prepd. from diazotized benzidine,  $CuSO_4$  and  $KCN$ , using  $C_6H_6$  to prevent froth. E in  $C_6H_6$  with  $MgMeI$  gave D. E in  $C_6H_6$  with  $MgC_2H_5Cl$  gave a reddish brown mass of 4,4'-*diphenyldiacetyldiphenyl*, m. 224°.

H. E. WILLIAMS

**Alcohols of the hydroaromatic and terpene series. III. Isopulegol.** ROBERT H. PICKARD, HAROLD HUNTER, WM. LEWCOCK and HANNAH S. DE PENNINGTON. *J. Chem. Soc.* 117, 1248–63(1920).—*l*-Isopulegol was isolated in the following manner: the alcs. obtained by hydrolyzing the condensation products of citronellaldehyde and  $Ac_2O$  were heated at 110° for 12 hrs. with phthalic acid. The product was dissolved in dil. aq.  $Na_2CO_3$  and then extd. with  $Et_2O$ .  $MgCl_2$  was then added and the pasty mass was strained and crystd. 8 times from aq. alc., yielding *magnesium l-isopulegyl phthalate* (A), m. 111°, sol. in  $CHCl_3$  or  $Me_2CO$ . With  $HCl$  it yields *l-isopulegyl hydrogen phthalate*, prisms from glacial  $AcOH$ , m. 106°. The *strychnine salt* was prepd., long needles, 205°, from abs. alc. *l-Isopulegol*, b. 88°, is obtained by hydrolyzing the phthalate with excess of  $NaOH$  in alc. It may be reduced in  $H_2O$  with gum arabic and  $Pd$  to *l*-menthol. By salting the mother liquors from which A was secured, using  $MgCl_2$ , a very sol.  $Mg$  salt was obtained and when this was hydrolyzed it yielded *d-α-isopulegol*. The esters of isopulegol formed from aliphatic acids were prepd. by the action of the requisite acid chloride on a soln. of isopulegol in  $C_6H_6N$ , except in the case of the acetate, propionate valerate and heptoate, where the acid anhydrides were employed. Tables are given showing the b. p., density, and index of refraction of 12 esters.

H. E. WILLIAMS

**Substituted quaternary azonium compounds containing an asymmetric nitrogen atom. III. Resolution of phenylmethylethylazonium, phenylbenzylpropylazonium and phenylbenzylallylazonium iodides into optically active components.** BAWA K. SINGH. *J. Chem. Soc.* 117, 1202–14(1920).—S. continued the work reported previously (*C. A.* 7, 2384; 8, 3782). Phenylmethylethylazonium *d-α*-bromocamphor- $\beta$ -sulfonate (A) was prepd. by adding the azonium iodide to *Ag d-α*-bromocamphor- $\beta$ -sulfonate in  $MeOH$ .  $AgI$  was formed and also crystals of A, m. 152–3°. *dl*-Phenylmethylethylazonium picrate (B) was obtained from the *dl*-azonium iodide, yellow prisms, m. 110–1°, sol. in  $Me_2CO$ , less sol. in  $MeOH$ ,  $EtOH$ , or  $CHCl_3$ . The *l*-form of B, prepd. by adding the bromocamphorsulfonate of the *l*-base in alc. to picric acid in alc., m. 114–5°. A mixt. of the *dl*- and *l*-picrate m. 107–8°. It is very sol. in  $Me_2CO$ , less so in  $MeOH$ ,  $EtOH$ ,  $CHCl_3$  and in  $H_2O$ , insol. in  $Et_2O$ . *asym*-Phenylpropylhydrazine (C) was prepd. by dissolving  $Na$  in  $PhNHNH_2$  at 18° under diminished pressure and treating the resulting mass with  $PrBr$  (Michaelis, *Ber.* 30, 2815 (1897)). The secondary hydrazine was purified by converting it into the  $HCl$  compd. This

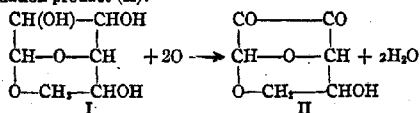
product, with  $K_4Fe(CN)_6$ , gave *phenyl- $\alpha$ -propylhydrazine hydroferrocyanide*, turns violet-blue in the air and is readily sol. in  $H_2O$ . C, with  $C_2H_5I$ , gave *dl-phenylbenzylpropylazonium iodide* (D), m.  $125^\circ$  (decompn.), readily sol. in MeOH, less so in EtOH. When PrI interacted with phenylbenzylhydrazine, D was not formed, but a product appeared which seemed to be a hydriodide. *dl-Phenylbenzylpropylazonium chloride*, m.  $145^\circ$ , was obtained as in the case of the iodide. The *chloroplatinate*, m.  $151^\circ$ , and the *chloroaurate*, m.  $132^\circ$ , were also prepd. D and Ag *d*-camphor- $\beta$ -sulfonate gave *phenylbenzylpropylazonium d-camphor- $\beta$ -sulfonate* (E), m.  $185^\circ$ , very sol. in MeOH, EtOH,  $Me_2CO$ , and  $CHCl_3$ , and when this was subjected to fractional pptn. by dissolving in MeOH and adding  $Et_2O$ , it was resolved into the *l*- and *d*-forms of E. *Phenylbenzylpropylazonium d- $\alpha$ -bromocamphor- $\beta$ -sulfonate* was prepd. by adding the azonium iodide to the necessary Ag salt. The product crystd. from  $Et_2O$  m.  $144^\circ$ , is sol. in MeOH, less so in EtOH,  $Me_2CO$ , and  $EtOAc$ . From this salt was prepd., using alc. and then  $Et_2O$ , *l*-phenylbenzylpropylazonium *d- $\alpha$ -bromocamphor- $\beta$ -sulfonate*. C and MeI gave *dl*-phenylmethylpropylazonium iodide (F), colorless prisms, m.  $106-7^\circ$ , from alc. and  $Et_2O$ , sol. in MeOH, EtOH and  $H_2O$ , but insol. in  $C_6H_6$ . By allowing PrI to act on phenylmethylhydrazine, the corresponding azonium iodide could not be obtained but  $NH_4I$  was isolated. When PrI acted on C, the corresponding azonium compd. also could not be obtained, but  $NH_4I$  was also obtained.  $C_2H_5I$  and  $PhNEtNH_2$  in  $Et_2O$  gave colorless prisms, m.  $107-8^\circ$  from alc. and  $Et_2O$ , of *dl-phenylethylallylazonium iodide*. The *chloroplatinate*, m.  $161^\circ$ , is an orange ppt. sol. in cold MeOH, more readily in hot, insol. in  $H_2O$ ,  $Me_2CO$ , EtOH,  $C_6H_6$  or  $Et_2O$ . *asym-Phenylallylhydrazine* resulted when  $C_6H_5NHNHNa$  interacted with allyl bromide (Michaelis and Claessen, *Ber.* 22, 2234 (1889)). The hydrochloride m.  $149-50^\circ$ , not  $137^\circ$ . When this compd. was treated with  $C_2H_5I$ , colorless prisms, m.  $115-6^\circ$ , of *dl-phenylbenzylallylazonium iodide*, were formed, very sol. in MeOH, less sol. in EtOH, sparingly sol. in  $H_2O$  or  $C_6H_6$ . *Phenylbenzylallylazonium d-camphor- $\beta$ -sulfonate* was prepd. in the usual way and is very sol. in MeOH, EtOH or  $Me_2CO$ . This yielded the *d*-rotatory compd., m.  $147^\circ$ , when treated with MeOH and  $Et_2O$ .

H. E. WILLIAMS

Pelletierine and methylpelletierine. GEORGES TANRET. *Bull. soc. chim.* 27, 612-24 (1920); cf. C. A. 14, 2336.—The same article is given in greater detail.

R. CHESTER ROBERTS

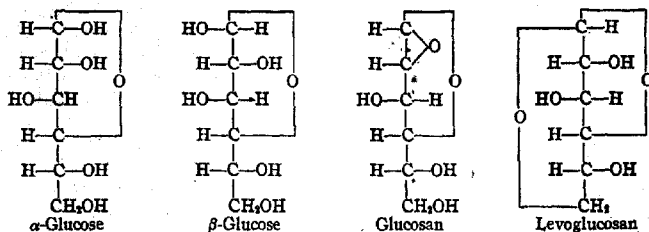
The constitution of levoglucosan. AMÉ PICET and MARC CRAMER. Geneva. *Helvetica Chim. Acta* 3, 640-4 (1920).—Levoglucosan is given the formula (I) from a study of its oxidation product (II):



II does not reduce Fehling soln., gives a neutral reaction to litmus, dissolves in  $NH_3$ , but does not combine with it, forms a *dihydrazone* with  $PhNHNH_2$ , small needles from hot water, m.  $154-5^\circ$ , combines with  $o-C_6H_4(NH_2)_2$  in AcOH or hot alc., forming a yellow, cryst. compd. with basic properties, forms a cryst. acetate with  $Ac_2O$ . These reactions point to a diketone, closed-ring compd. of the type II. P. and C. discuss other formulas that have been suggested by other workers. R. CHESTER ROBERTS

The configuration of  $\alpha$ - and  $\beta$ -glucose. AMÉ PICET. Geneva. *Helvetica Chim. Acta* 3, 649-52 (1920).— $\alpha$ - and  $\beta$ -Glucose are stereoisomers and can be considered as compds. with the hydrofuran nucleus, with the HO group attached on opposite sides to the first C atom of the chain. Tanret states that  $\alpha$ -glucose is slowly converted into  $\beta$ -glucose when kept at  $110^\circ$ , showing the latter to be the more stable modification.

Böecklen (*C. A.* 8, 95) accounts for the formation of 5-membered rings when  $\alpha$ -glucose is treated with  $H_2BO_3$  because the OH radicals are adjacent to each other. The elec. cond. of  $H_2BO_3$  is increased by the presence of certain alcs. and  $\alpha$ -glucose exhibits this same property to a much greater extent than  $\beta$ -glucose. P. does not think these two views of T. and B. constitute decisive exptl. proofs of the structures. P. derives the structure of  $\alpha$ - and  $\beta$ -glucose from the anhydrides which they will form.  $\alpha$ -Glucose gives glucosan and  $\beta$ -glucose gives levoglucosan. The graphic structures for all four will illustrate their similarities and differences:



R. CHESTER ROBERTS

**Glucosides.** VII. The constitution of amygdalin. P. KARRER, C. NÄGELI AND L. LANG. *Helvetica Chim. Acta* 3, 573-84(1920); cf. *C. A.* 14, 1973.—The nature of the disaccharide in amygdalin has never been detd. K. has asked the question if it could be cellobiose, and answered it by synthesizing the corresponding acid compd., and has shown that the products are not identical. The question still remains unanswered as far as the nature of the disaccharide is concerned.  $\beta$ -Tetracetyl-d-glucosido-d,l-mandelyl chloride, obtained by the action of 5 g.  $PCl_5$  upon 4 g. of the acid, thin needles, m. 117-9°. With  $NH_3$  the amide is formed, powder, m. about 100°. Heptaacetylcellosido-d,l-mandelic acid, prepd. by heating 12 g. acetobromocellobiose, 4.4 g. Ag mandelate and 100 cc.  $C_{10}H_{12}$  to boiling, long needles, m. 179-82°,  $[\alpha]_D^{20}$  -44°. It does not reduce Fehling soln. Heptaacetylcelloside, by warming 3 g. acetobromocellobiose, 3 g.  $Ag_2CO_3$  and 50 cc. alc. for 5 min., needles, m. 184°,  $[\alpha]_D^{20}$  -24.76°. Upon hydrolysis this yields ethylcelloside, a hygroscopic powder, which reduces Fehling soln. on boiling,  $[\alpha]_D^{20}$  -9.55°. The heptaacetylamygdalinic acid is an amorphous material (Schiff, *Ann.* 154, 337(1870)), m. unsharply at 60-100°, and having  $[\alpha]_D^{20}$  21°. C. J. WERT

**Polysaccharides.** I. Methylation of starch. P. KARRER. Zurich. *Helvetica Chim. Acta* 3, 620-5(1920).—K. attempts to show that the assumption made by Hess (*Z. Elektrochem.* 26, 232(1920)) that cellulose is built up of simpler units by means of residual affinities is unnecessary. He believes that the starch crystalloid is made up of starch mols. bound together in the same way as Ag mols. are in the Ag particle. In the process of methylation the starch is rendered sol., a product containing 2 OMe groups being sol. in  $H_2O$ , alc. and  $CHCl_3$ ; during this process the starch reaction with I decreases from a blue to a pale yellow. Simultaneously with the methylation, the crystalloid is depolymerized, so that in the methylated product we have the simple mol. Exptl. details will be given later. Also in *Paper* 27, 24 (1920). C. J. WERT

**Diazotization of polynitroanilines.** EMIL MISSLIN. *Helvetica Chim. Acta* 3, 626-39(1920).—The introduction of  $NO_2$  groups into  $PhNH_2$  increases the difficulty with which it is diazotized. M. has developed a method whereby this is easily carried out. The amine is dissolved in AcOH and carefully treated, in the cold, with  $H(NO_3)SO_4$  or with  $NaNH_2$  in 100%  $H_2SO_4$ . In the case of  $(O_2N)_3C_6H_2NH_2$ , as much monohydrate should be used as of AcOH. 4,2,6- $Cl(O_2N)_3C_6H_2NH_2$  is easily diazotized in this way, and yields with  $\beta$ - $C_{10}H_7OH$  steel-blue crystals from AcOH, or dark reddish brown

crystals from  $\text{PhNO}_2$ , having the compn.  $\text{C}_{18}\text{H}_{10}\text{O}_2\text{N}_4\text{Cl}$ , sol. in concd.  $\text{H}_2\text{SO}_4$  with a pure violet-blue color, from which  $\text{H}_2\text{O}$  ppts. it unchanged. The dye from  $\alpha\text{-C}_{10}\text{H}_7\text{OH}$  forms fine, brownish yellow needles with 1 mol.  $\text{AcOH}$ . Typical for this dyestuff is its soly. in dil.  $\text{NaOH}$  with a deep blue color;  $\text{KCl}$  or  $\text{NaCl}$  soln. ppts. the corresponding salt in crystal violet-like, glistening needles. Other dyestuffs were prepd. 4,2,6- $\text{HO}_2\text{S}(\text{O}_2\text{N})_2\text{C}_6\text{H}_2\text{NH}_2$  was diazotized by soln. in cold  $\text{H}(\text{NO})\text{SO}_4$  and addition of ice till a 70%  $\text{H}_2\text{SO}_4$  resulted.  $\beta\text{-C}_{10}\text{H}_7\text{OH}$  gave an orange-brown cryst. ppt., which dyes wool in slightly acid bath an intense orange, or a carmine-red in the presence of  $\text{CuSO}_4$ . The  $\alpha\text{-C}_{10}\text{H}_7\text{OH}$  dyestuff is a brownish yellow powder, dyeing wool a yellowish brown color, which is sensitive to alkali, and is changed to a deep blue by dil.  $\text{Na}_2\text{CO}_3$ . 2,4,6- $(\text{O}_2\text{N})_3\text{C}_6\text{H}_2\text{NH}_2$  was diazotized in  $\text{AcOH}$  with an excess of  $\text{H}_2\text{SO}_4\cdot\text{H}_2\text{O}$ . The combination with  $\beta\text{-C}_{10}\text{H}_7\text{OH}$  is a greenish blue, glistening powder from  $\text{AcOH}$ , or reddish brown from  $\text{PhNO}_2$ , sol. in concd.  $\text{H}_2\text{SO}_4$  with a pure blue-violet color. The  $\alpha\text{-C}_{10}\text{H}_7\text{OH}$  dyestuff is yellowish brown, sol. in concd.  $\text{H}_2\text{SO}_4$  with a pure blue-violet color, and in dil.  $\text{NaOH}$  with a pure blue color, from which  $\text{NaCl}$  and  $\text{KCl}$  ppt. the blue, glistening salts. 3,2,4,6- $\text{HO}(\text{O}_2\text{N})_2\text{C}_6\text{H}_2\text{NH}_2$  yields a pure orange-yellow diazo salt.  $\beta\text{-C}_{10}\text{H}_7\text{OH}$  gives a brown-black ppt., which dissolves in concd.  $\text{H}_2\text{SO}_4$  to a pure blue color, and in dil.  $\text{NaOH}$  with a Bordeaux color. Slight warming of the alk. soln. destroys the dyestuff. The  $\text{MeO}$  deriv. was also easily diazotized, and gave a dyestuff,  $\text{C}_{17}\text{H}_{11}\text{O}_4\text{N}_4$ , reddish brown glistening crystals. The  $\text{EtO}$  deriv. gave a dyestuff,  $\text{C}_{19}\text{H}_{13}\text{O}_4\text{N}_4$ , orange-brown needles from  $\text{PhNO}_2$ . 2,4,6,1,3- $(\text{O}_2\text{N})_4(\text{NH}_2)_2\text{C}_6\text{H}_2$  gives a deep orange diazo salt, which couples with  $\beta\text{-C}_{10}\text{H}_7\text{OH}$  to give a dark reddish brown ppt., sol. in concd.  $\text{H}_2\text{SO}_4$  with a pure blue color. It was not decided whether the tetra- $\text{NO}_2$  deriv. could be diazotized.

C. J. WEST

**Reactions of  $\alpha$ -campholide and bromocampholic esters.** H. RUPF AND A. JÄGGI. *Helvetica Chim. Acta* 3, 654-68(1920).— $\alpha$ -Campholide (A) was prepd. by the reduction of camphoric anhydride with  $\text{H}$  at  $220\text{--}40^\circ$ , using  $\text{Ni}$  as the catalyst. As a by-product a hydrocarbon,  $\text{C}_{16}\text{H}_{16}$ , b.  $120\text{--}32^\circ$ , was isolated. Bromocampholic acid was best prepd. by satg. A in  $\text{AcOH}$  with  $\text{HBr}$ . The chloride,  $\text{C}_{10}\text{H}_{15}\text{OClBr}$ , prepd. by the use of  $\text{SOCl}_2$ , compact monoclinic crystals, b<sub>11</sub>  $142^\circ$ , m.  $57.5^\circ$ . Amide,  $\text{C}_{10}\text{H}_{15}\text{ONBr}$ , by the action of dry  $\text{NH}_3$  gas upon the chloride, fine needles, m.  $207^\circ$ . Anilide, needles, m.  $142^\circ$ . Phenylhydrazide,  $\text{C}_{18}\text{H}_{23}\text{ON}_2\text{Br}$ , fine needles, m.  $151\text{--}1^\circ$ . Methyl ester, oil with an odor of pepper, b<sub>13</sub>  $139^\circ$ . Ethyl ester, b<sub>13</sub>  $142\text{--}3^\circ$ , m.  $9\text{--}10^\circ$ . Phenyl ester, large plates, b<sub>11</sub>  $204^\circ$ , m.  $46\text{--}7^\circ$ . The  $\text{Me}$  ester reacts with  $\text{PhCH}_2\text{MgCl}$  to form a compound, 1,2,2-trimethyl-3-phenethylcyclopentyl benzyl ketone,  $\text{C}_{24}\text{H}_{38}\text{O}$ , large, glistening needles, m.  $119^\circ$ , b<sub>14</sub>  $245\text{--}50^\circ$ . The  $\text{Et}$  ester, heated with  $\text{PhNH}_2$ ,  $\text{PhNMe}_2$ , or quinoline, gave A, thus losing 1 mol.  $\text{EtBr}$ .  $\text{MeOH-KOH}$ , or better  $\text{EtONa}$ , gave ethyl 1,2,2-trimethyl-3-methylcyclopentane-1-carboxylate,  $\text{C}_{12}\text{H}_{20}\text{O}_2$ , oil, b<sub>12</sub>  $93^\circ$ ,  $[\alpha]_D^{20}$   $26.73^\circ$ ,  $d_4^{20}$   $0.9466$ ,  $M_\alpha$   $1.45575$ ,  $M_D$   $1.45773$ ,  $M_\beta$   $1.46486$ ,  $M_\gamma$   $1.47006$ .  $\text{HBr}$  regenerates the original ester.  $\text{Br}$  in  $\text{CHCl}_3$  gives a compound,  $\text{C}_{12}\text{H}_{19}\text{O}_2\text{Br}$ , needles or leaflets, m.  $149^\circ$ . 1,2,2-Trimethylcyclopentane-1,3-methanol, prepd. by reducing A with  $\text{Na}$  and alc., fine, glistening needles, b<sub>12</sub>  $151\text{--}4^\circ$ , m.  $130^\circ$ .  $\alpha$ -Camphidone (Tafel and Eckstein, *Ber.* 34, 3274(1901)) is conveniently prepd. from A by heating with  $\text{ZnCl}_2\text{-NH}_4\text{OH}$  12 hrs. in a sealed tube at  $150\text{--}60^\circ$ . By heating the glycol in the same way, camphidine is formed.

C. J. WEST

**Behavior of glycol and its derivatives and homologs upon electrochemical oxidation.** FR. FICHTER AND MAX SCHMID. *Helvetica Chim. Acta* 3, 704-14(1920).— $\text{H}_2\text{NCH}_2\text{CO}_2\text{H}$ , upon oxidation by the elec. current, yields  $\text{NH}_3$ ,  $\text{HCHO}$  and  $\text{CO}_2$ . The 1st 2 react to form a mixt. of amines, which was isolated as chlorides. The ratio of  $\text{CO}_2\text{:NH}_3$  varied from 1:1.2 to 1:0.712, depending upon the conditions of the electrolysis.  $\text{AcNHCH}_2\text{CO}_2\text{H}$  and  $\text{AcNHCH}_2\text{CO}_2\text{Na}$  gave a higher yield of  $\text{CO}_2$  than  $\text{NH}_2\text{CH}_2\text{CO}_2\text{H}$ ,  $\text{NH}_3$  and  $\text{AcOH}$ , as well as  $\text{HCHO}$  and  $\text{HCO}_2\text{H}$ , are also formed.

$(\text{HO}_2\text{CCH}_2)_2\text{NH}$  decomp. into  $4\text{CO}_2$ ,  $\text{NH}_3$  and  $2\text{H}_2\text{O}$ .  $\text{MeN}(\text{CH}_2\text{CO}_2\text{H})_2$  yields  $\text{MeNH}_3$ ,  $4\text{CO}_2$  and  $2\text{H}_2\text{O}$ .  $\text{NH}_2\text{CMe}_2\text{CO}_2\text{H}$  decomp. into  $\text{NH}_3$ , acetone and  $\text{CO}_2$ .  $\alpha$ -*p*-Tolylsulfonamidoisobutyric acid, long white needles, m.  $147^\circ$ .  $\gamma$ -Benzenesulfonamidoisobutyric acid, needles, m.  $144^\circ$ . *N*-Methyl- $\alpha$ -benzenesulfonamidoisobutyric acid, needles, m.  $171^\circ$ .  $\beta$ -Alanine, upon electrolysis in  $\text{H}_2\text{SO}_4$ , gave  $\text{NH}_3$ ,  $\text{CO}_2$ ,  $\text{AcOH}$  and  $\text{HCHO}$ . *Benzoylalanine*, glistening leaflets, m.  $119^\circ$ . C. J. WEST.

**Isomerization and polymerization of methyl thiocyanate.** J. GILLIS. Univ. Amsterdam. *Rec. trav. chim.* 39, 330-8 (1920).—In repeating the expts. of Walden (*Z. physik. Chem.* 55, 297 (1906)) G. observed that at  $130^\circ$  the influence of various compds. dissolved in  $\text{MeSCN}$  (A) is different. Except with  $\text{CdI}_2$  and  $\text{H}_2\text{SO}_4$  the catalytic action in favor of isomerization and of polymerization is minimum. The degree of purity of the solvent has a great influence on the transformations of A. G. demonstrated that A is already isomerized appreciably at  $130^\circ$  as shown by thermal analysis and refractometric analysis of the 1st portions from the distn. Likewise the polymerization of A may be distinctly observed at  $130^\circ$ . This was detd. by measurements of the mol. wt. of the polymer formed. The b. p. const. of  $\text{MeNCS}$  (B) is in agreement with the const. deduced by Trouton's rule. In following the behavior of A and the polymerization at different temps. it was found that the isomerization is a practically complete reaction, but that it is always accompanied by the formation of the trimer. The addition of foreign substances catalyzes the isomerization and polymerization differently. Thus  $\text{CdI}_2$  accelerates the formation of  $\text{MeNCS}$ , while  $\text{H}_2\text{SO}_4$  favors the formation of  $(\text{MeSCN})_3$ . It seems that in the long run the polymerization ought to surpass the isomerization, which accounts for the failure of Bremer's attempt (*C. A.* 5, 876) to prep. the Me mustard oil from its isomer. E. J. WITZEMANN

**The action of an alcohol solution of potassium hydroxide on ketones.** V. The action of alcoholic potassium hydroxide on *p*-hydroxy- and *p*-ethoxybenzophenone and on their *p*-chloro and *p*-bromo derivatives. P. J. MONTAGNE. Univ. Leyden. *Rec. trav. chim.* 39, 339-49 (1920).—Previous work (*C. A.* 3, 421; 7, 1178; 11, 1176, 3032) has shown that the aptitude of the CO group in  $\text{BzPh}$  derivs. to undergo reduction depends on the nature and the position of the substituents in the ring. The results described in this paper relate to the influence of OH and OEt in the *p*-position as well as that of Cl and Br in the *p*-position in the other ring. The results show that 4-HO- $\text{C}_6\text{H}_4\text{COPh}$  undergoes scarcely any reduction in  $\text{EtOH-KOH}$  even on boiling for 6 days. 4-Hydroxy-4'-chlorobenzophenone was recovered unchanged after boiling for 2 days and again after boiling 6 days. The Br deriv. gives the same result. The 4-EtOC $_6\text{H}_4$ -COPh was heated for 4 days and was recovered unchanged. On repeating the expt. and boiling for 6 days the material was converted into 4-ethoxybenzohydrol. The influence of the OEt group is therefore much weaker than that of the OH group. 4-Ethoxy-4'-chlorobenzophenone is largely converted into 4-ethoxy-4'-chlorobenzohydrol after boiling 2 days only. Some of the unchanged compd. is recovered as well as some yellow crystals that m.  $109^\circ$ . After boiling 3.5 days only the hydrol and the compd. m.  $109^\circ$  were recovered. If boiled 6 days none of the compd. m.  $109^\circ$  was obtained and besides the hydrol a compd. that m.  $130^\circ$  was obtained. Boiling this compd., m.  $130^\circ$ , with  $\text{KOH-EtOH}$  again for 6 days raised its m. p. to  $131.5^\circ$  and it was identified as  $(4\text{-EtOC}_6\text{H}_4)_2\text{CO}$ . It is evident that the product that m.  $109^\circ$  is a mixt. of  $\text{CO}(\text{C}_6\text{H}_4\text{OEt})_2$  and  $\text{ClC}_6\text{H}_4\text{COC}_6\text{H}_4\text{OEt}$ . The same group of observations was made with the Br deriv. These expts. show that the Cl and Br atoms in the *p*-position have the opposite effect to that of the OEt group. This is the first instance of a replacement of a Cl or Br atom by an EtO group in a halogenated benzophenone by boiling with alc. KOH. The constitution of 4-EtOC $_6\text{H}_4$ COPh was proved by transforming it into 4-HOC $_6\text{H}_4$ COPh, of which the constitution had already been detd. 4-Chloro-4'-ethoxybenzophenone was prepd. from 4-ClC $_6\text{H}_4\text{COCl}$  and  $\text{PhOEt}$  with  $\text{AlCl}_3 + \text{CS}_2$ .



The position of the EtO group was proved by transforming it into a  $\text{ClC}_6\text{H}_4\text{COC}_6\text{H}_4\text{OH}$  identical with that obtained from 4-chloro-4'-aminobenzophenone. Full exptl. details are given. VI. The action of alcoholic potash on the iodobenzophenones (and the iodo-benzohydrols). *Ibid* 350-7.—Previous expts. (cf. preceding refs.), have shown that in the halogenated benzophenones the halogen is replaced entirely or mostly by H by the action of KOH-EtOH and that this replacement depends on the nature and the position of the halogen atom. The quantity replaced increases in the order Cl, Br, I. As for the influence of position the expts. with chloro- and bromobenzophenones have shown that the halogen in the *o*-position is mostly replaced, in the *m*-position largely replaced, while in the *p*-position it is least affected. The results with mono-iodobenzophenones are given in detail in this paper. 2- and 4-Iodobenzophenone are both transformed entirely, or nearly so, into benzohydrol. Under the same conditions the 3-isomer remains unattacked in part and is partly probably transformed into benzohydrol. When a group is introduced into 4- $\text{IC}_6\text{H}_4\text{COPh}$  which has a moderating influence on the reduction of the CO this influence seems to endure: 4-iodo-4'-ethoxybenzophenone is converted into 4- $\text{EtOC}_6\text{H}_4\text{COPh}$ . M. has examined the action of EtOH-KOH on the chloro- and bromobenzohydrols. It was found that while the halogen atom in benzophenones is replaced to a greater or less extent depending on the nature of the halogen atom and its position, with the chloro- and bromobenzohydrols on the contrary, not a trace of halogen is eliminated. The expts. with 3- and 4-iodobenzohydrol, however, showed an elimination of I to some extent. In this respect the I derivs. differ in their behavior from the Cl and Br derivs.

E. J. WITZEMANN

The action of an alcoholic solution of caustic potash in the presence of zinc dust on the brominated benzophenones and on some of their derivatives. P. J. MONTAGNE. Univ. Leyden. *Rec. trav. chim.* 39, 483-91(1920).—Zagoumenny (*Ann.* 184, 175 (1877)) reduced BzPh to benzohydrol in EtOH-KOH soln. with Zn dust, which Cohen (*C. A.* 13, 2504) and M. (*Rec. trav. chim.* 25, 402(1906)) have found gives nearly quant. results. 5 other references for its use are given. The opinion that benzophenones in general are reduced to benzohydrols by this method appears to be well founded, especially since M. (*Rec. trav. chim.* 24, 115(1905)) obtained the hydrol of (*p*- $\text{ClC}_6\text{H}_4$ )<sub>2</sub>CO in this way. However, when applied to (*p*- $\text{BrC}_6\text{H}_4$ )<sub>2</sub>CO the 2 Br atoms were displaced by H and benzohydrol was obtained. On treating (*p*- $\text{BrC}_6\text{H}_4$ )<sub>2</sub>CHOH in the same way (cf. following abstr.), benzohydrol was obtained. M. has extended these expts. to the other bromobenzophenones and their derivs. in order to see if Br is also replaced by H independently of its position and the presence of other groups. The results show that this is the case in all of these compds. examd. The reaction does not always take place smoothly, since tar is formed and sometimes in considerable amts. The expts. were done as follows: the benzophenone was heated in a flask with the EtOH-KOH soln. (whether it dissolved or not) and the Zn dust was added gradually with continual agitation. After all Zn dust had been added, heating was continued about 1 hr. with frequent agitation. The mixt. was then cooled, filtered, then  $\text{H}_2\text{O}$  was added, and the EtOH evapd. This gives residue A, the treatment of which is described for each case, but generally consisted in extg. it with petr. ether (b. 50-80°), leaving the tar as a residue, evapg., distg. the crystals of benzohydrol at reduced pressure and identifying the product by its m. p. after crystg. again. 2-, 3- and 4-bromo, 2,4-, 2,6-, 2,4', and 4,4'-dibromo-, 2,4,6-tribromo-, 2-bromo-4'-ethoxy-, 4-bromo-4'-ethoxy-, 4-bromo-3'-amino-, 4-bromo-4'-amino-, and 4,4'-dibromo-3,3'-diaminobenzophenones were treated in this way and in each case gave the corresponding benzohydrol deriv. with Br substituted with H. The crystallographic data and the detn. of the constitution of 2-bromo-4'-ethoxybenzophenone are given.

E. J. WITZEMANN

The action of an alcoholic solution of caustic potash in the presence of zinc dust on the benzohydrols and on some of their derivatives. P. J. MONTAGNE. *Rec. trav.*

*chim.* 39, 492-4(1920).—In the course of previous work (M. and v. Charante, *C. A.* 7, on the action of EtOH-KOH on brominated benzophenones and benzohydrols 1178) it was found that the Br atoms in the former are replaced by H while the latter are stable. When Zn dust is added to the mixt. this difference no longer appears. The Br atom is replaced by H in both series of compds. In the reaction on bromobenzophenones tar is formed, but this does not occur with the corresponding benzohydrols. The expts. were done as follows: the bromobenzohydrol was heated with 10% KOH in EtOH, the Zn dust was added gradually and the mixt. was heated 1.5 hrs. longer on the H<sub>2</sub>O bath. After cooling the liquid was filtered, treated with H<sub>2</sub>O and the EtOH evapd., giving a ppt. which was treated as described in the preceding abstr. 2-, 3- and 4-bromo-, 4,4'-dibromo-, 4-bromo-3-amino-, 4-bromo-3'-amino-, 4,4'-dibromo-, 3,3'-diamino-, and 4-bromo-4'-ethoxybenzohydrol were treated in this way and gave in each case the corresponding benzohydrol with all Br substituted with H.

E. J. WITZEMANN

The replacement of substituents in the benzene ring. III. A. F. HOLLEMAN. Amsterdam. *Rec. trav. chim.* 39, 435-80(1920).—In the 1st paper (*C. A.* 10, 1508) H. discussed the complexity of the problem and gave evidence by the work of his pupils, de Mooy and ter Weel. Meanwhile H. has restricted the exptl. study to aromatic compds., having only Cl and NO<sub>2</sub> as substituents because a certain number of them have already been studied. Theoretically, there are 91 aromatic compds. containing only Cl and NO<sub>2</sub>. Of these 55 are known. All of the 12 compds. containing only Cl are known. Omitting PhCl in which the Cl reacts with difficulty the velocity of reaction of the dichlorobenzenes with NaOMe was studied by De Mooy (*C. A.* 10, 1509) and that of the tri-Cl derivs. by H. (*C. A.* 13, 569). Compds. containing only NO<sub>2</sub> are 12 in number, of which 7 are known. The NO<sub>2</sub> in PhNO<sub>2</sub> cannot be substituted but Steger (*Rec. trav. chim.* 18, 20(1899)) detd. the reaction velocity of *o*- and *p*-(O<sub>2</sub>N)<sub>2</sub>-C<sub>6</sub>H<sub>4</sub> with EtONa and MeONa. Of the compds. with one Cl and 1 or more NO<sub>2</sub> groups there are 19, of which 11 are known. The velocity of reaction of most of these has been studied by de Mooy, ter Weel and Lulofs (*Rec. trav. chim.* 20, 292(1901)). Of compds. containing 2 Cl atoms and NO<sub>2</sub> groups there are 26, of these 15 are known, of which the reaction velocity of dichloromononitro derivs. was studied by De Mooy. Of the 15 tri-Cl derivs. containing 1, 2 or 3 NO<sub>2</sub> groups, 8 are known imperfectly and the reaction velocity has not been studied. O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Cl is known, but its reaction velocity has not been measured. The gaps are therefore still considerable. In this paper the results of de Hollander on 11 dichlorodinitrobenzenes are given. The dichlorodinitrobenzenes and their action with sodium methylate. A. J. DEN HOLLANDER.—Eight of the 11 dichlorodinitrobenzenes are described in the literature, although there was some confusion about the constitution of some of them. Their prepn. has been revised, the unknown ones have been prepd., and the constitutions of all established. I. *Prepn. of dichlorodinitrobenzenes*.—1,3,4,6-C<sub>2</sub>H<sub>2</sub>Cl<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub> (A) was previously prepd. by Körner, Nietzki and Schedler and Fries, and was obtained thus: *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> → *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Cl → *m*-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Cl → *m*-C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> → A. The 1,3,4,5-isomer (B) had been prepd. by Blanksma (*C. A.* 2, 1122) and Körner and Contardi (*C. A.* 8, 1761) thus: *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> → 3,5,4-Cl<sub>2</sub>(H<sub>2</sub>N)C<sub>6</sub>H<sub>2</sub>NO<sub>2</sub> → 3,5-Cl<sub>2</sub>C<sub>6</sub>H<sub>2</sub>NO<sub>2</sub> → B. The 1,3,2,5-isomer (C) had been prepd. by K. and C. thus: *p*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> → 3,5,4-Cl<sub>2</sub>(H<sub>2</sub>N)C<sub>6</sub>H<sub>2</sub>NO<sub>2</sub> → C. The 1,3,2,4-isomer (D) had been obtained by K. and C. thus: 1st 3 stages as with B → 3,5-Cl<sub>2</sub>C<sub>6</sub>H<sub>2</sub>NH<sub>2</sub> → 3,5-Cl<sub>2</sub>C<sub>6</sub>H<sub>2</sub>NHAc → 3,5,4-Cl<sub>2</sub>(O<sub>2</sub>N)C<sub>6</sub>H<sub>2</sub>NHAc → free NH<sub>2</sub> deriv. → 2,6-Cl<sub>2</sub>C<sub>6</sub>H<sub>2</sub>NO<sub>2</sub> → D. The 1,4,2,6-isomer (E) was obtained by Jungfleisch (*Ann. chim. phys.* [4], 13, 259) and Ullmann and Sané (*C. A.* 6, 864) by 2 methods; that of J. consisted in nitrating *p*-Cl<sub>2</sub>C<sub>6</sub>H<sub>4</sub>. The mother liquors from E obtained by J.'s method contain the 1,4,2,3-isomer (F), which is sepd. by

fractional crystn. from EtOH and was the  $\beta$ -isomer obtained by J. The constitution of F was established by synthesizing it from 3,6,2-Cl<sub>3</sub>(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>NHAc. The 1,4,2,5-isomer (G) was obtained as follows: *p*-Cl<sub>2</sub>C<sub>6</sub>H<sub>4</sub> was nitrated (de Mooy, C. A. 10, 1509). The 2,5-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NO<sub>2</sub> obtained was reduced with Fe powder and HCl. The Ac deriv. of the NH<sub>2</sub> deriv. was nitrated thus: 300 cc. HNO<sub>3</sub> (d. 1.51) were treated gradually with 100 g. of the anilide at -5° to 0°. The soln. was poured on crushed ice, filtered and washed. The product was a mixt. of 2 isomers which were sepd. by Beilstein and Kurbatov's method (*Ann.* 196, 221(1879)) by extg. in a Soxhlet app. with C<sub>6</sub>H<sub>6</sub> in which the desired isomer is insol. The 2,5,4-Cl<sub>3</sub>(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>NHAc was sapond. and nitrated by the method of Körner and Contardi (*l. c.*). G was obtained in this way and by 2 other methods, and described as faintly yellow needles that m. 119°. The 1,2,4,5-isomer (H) had previously been prepd. by Blanksma (*Rec. trav. chim.* 21, 419 (1902)) by substituting Cl for NH<sub>2</sub> by diazotizing, etc., in *o*-ClC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> and then nitrating. The 1,2,3,4-isomer (I) previously unknown was obtained by the following steps: *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> → *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHAc → 2,3-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NHAc → the free NH<sub>2</sub> deriv. → 2,3-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>Cl → I. The I obtained by this and another method seps. as long colorless needles from ligroin, m. 97°. The 1,2,3,5-isomer (J) was obtained by the nitration of 2,3- and 3,4-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NO<sub>2</sub> and of *o*-Cl<sub>2</sub>C<sub>6</sub>H<sub>4</sub>. The 1,2,3,6-isomer (K), previously known, proved to be very difficult to obtain. Several methods were tried, but the best results were obtained by starting with *m*-nitroaniline, which was nitrated as the Ac deriv. and subsequently sapond., giving 2,5-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NH<sub>2</sub> according to de Mooy (C. A. 10, 1509) and Ter Weel (C. A. 10, 1511). On chlorination the desired compd, 6,2,5-Cl(O<sub>2</sub>N)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>NH<sub>2</sub>, was a secondary product. Without sepg. the 2 isomers the mixt. was diazotized by Gattermann's method and the K sepd. by crystg. from EtOH, m. 60°. It is stated that not much K was obtained and it was not sufficiently studied. II and III. *Action of sodium methylate on the dichlorodinitrobenzenes*: The expts. developed the following qual. facts: (1) When a Cl atom is eliminated in the chloro-, nitro-, dichloronitro-, chlorodinitro-, or dichlorodinitrobenzenes (L) it is always removed from the *o*- or *p*-position with respect to a NO<sub>2</sub> group; the introduction of a 2nd NO<sub>2</sub> group into the chloronitro- and the dichloronitrobenzenes renders the Cl in the *m*-position with respect to the NO<sub>2</sub> already present inattacked. In L the Cl unattacked resists the action of a 2nd mol. of NaOMe if it is in the *m*-position with respect to the 2 NO<sub>2</sub> groups. In all other cases the 2 Cl atoms are replaceable successively by MeO. (2) When a NO<sub>2</sub> is eliminated from L it is always *o*- or *p*- to a Cl atom. (3) When in chlorodinitrobenzenes or in L the 2 NO<sub>2</sub> groups occur *m*- to one another it is always a Cl atom that is attacked. (In *sym*-ClC<sub>6</sub>H<sub>4</sub>(NO<sub>2</sub>)<sub>2</sub> the Cl is inactive.) (4) In the chloronitrobenzenes and in L the activity of the Cl atom is suppressed by the introduction of a 2nd NO<sub>2</sub> group if it is placed *m*- with respect to this Cl atom. One of 2 groups then become, capable of substitution. (5) In *m*-dinitro-, *m*-chloronitro- and *sym*-chlorodinitrobenzene as well as in *m*-chloronitro- and *sym*-dichloronitrobenzene, which are all inactive toward NaOMe, any of the inactive substituents present may be activated by the introduction of a Cl atom or a NO<sub>2</sub> group. However, if one of the substituents is eliminated it is always that which was last introduced. A similar series of quantitative conclusions is given which cannot be satisfactorily abstracted. General conclusions follow: (1) The activity of a substitutable Cl is increased considerably when one introduces a NO<sub>2</sub> group in the *p*-position. (2) The activity of a substitutable Cl is sensibly increased by the introduction of a NO<sub>2</sub> in the *o*-position. (3) The activity of a NO<sub>2</sub> group becomes greater by the introduction of a Cl atom; when this Cl atom is introduced in the *m*-position with respect to the NO<sub>2</sub> group it exercises a much greater influence. (4) It is probable that in 1,3,2,4-C<sub>6</sub>H<sub>3</sub>Cl<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub> it is the Cl atom in position 1 that is substitutable. Cf. the original for many tabulated and numerical data.

E. J. WITZERMANN

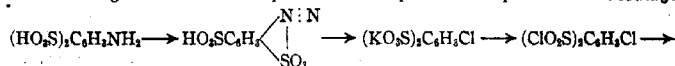
Some observations on chlorinated quinones. A. J. DEN HOLLANDER. Univ. Amsterdam. *Rec. trav. chim.* 39, 481-2(1920).—In the synthesis of 1,2,3,6- $C_6H_2Cl_4(NO_2)_2$ , d. H. attempted to obtain the 2,3-dichloro-1,4-quinone described by Peratoner and Genco (*Gazz. chim. ital.* 24, II, 394(1894)) and Olivieri and Tortorici (*Gazz. chim. ital.* 27, II, 584(1897)) but could not confirm their results. The 1st step was the prepn. of chloroquinol (A) from quinone according to Clark (*Am. Chem. J.* 14, 571(1892)). The oxidation of A to the corresponding quinone was done as follows: 50 g. A in 1260 cc.  $H_2O$  was treated, drop by drop, with 50 g.  $K_2CrO_4$  in 500 cc.  $H_2O$  + 50 cc. concd.  $H_2SO_4$  at  $0^\circ$ . The quinone was filtered off and when treated after purification according to the directions of P. and G. and O. and T. with dry  $HCl$  in  $Et_2O$  gave a mixt. of dichloroquinols which could not be sepd. and purified. When the mixt. was oxidized as above with  $H_2CrO_4$  at about  $75^\circ$  and then cooled 2,5-dichloro-1,4-quinone (m.  $161^\circ$ ) and 2,6-dichloro-1,4-quinone (m.  $120-1^\circ$ ) were obtained. No 2,3-dichloro-1,4-quinone was present and this compd. should, therefore, be considered as unknown.

E. J. WITZEMANN

Is the direct synthesis of urea under the influence of urease possible? TH. J. F. MATTAAR. Univ. Leyden. *Rec. trav. chim.* 39, 495-8(1920).—In chapter XI of his paper Barendrecht (*idem* 39, 73(1920); cf. C. A. 14, 748) describes a method by which he believes he demonstrated the synthesis of urea under the influence of urease from soy bean flour. The method of Posse (*Ann. inst. Pasteur* 30, 525(1920)) permits of testing this conclusion. This depends on the fact that xanthidrol with urea (0.001 g. per l. or more) ppts. dixanthylurea. M. observed that 2 cc. of a soln. containing 0.020 g. urea + 0.030 g.  $(NH_4)_2CO_3$  per l. gave a turbidity in 10 mins. and a heavy ppt. in 1 hr. B.'s expts. were repeated and the amts. of urea formed detd. by this method. The results are given in tables and show that under the conditions chosen by B. the synthesis of urea does not take place. His conclusion that he had synthesized urea under the influence of urease is not correct. This does not exclude the possibility of this synthesis but more favorable conditions will have to be found.

E. J. WITZEMANN

The sulfonation of metanilic acid. S. C. J. OLIVIER. Wageningen. *Rec. trav. chim.* 39, 499-504(1920).—The sulfonation of  $m\text{-H}_2\text{NC}_6\text{H}_4\text{SO}_3\text{H}$  was first done by Limpricht and Drebes (*Ber.* 9, 552(1876)) who obtained what they thought was 3,4- $(HO_2S)_2C_6H_3NH_2$ , since they thought they obtained  $o\text{-C}_6\text{H}_4(SO_3H)_2$  by diazotizing it. Zander showed (*Ann.* 198, 25(1879)) that the latter was a phenetoledisulfonic acid. In 1906 Schultz (*Ber.* 39, 3346(1906)) studied the constitution of the sulfonation products of metanilic acid and succeeded in removing the  $NH_2$  group from the dimetanilic acid (A) produced by converting it into the hydrazino compd. which was then boiled with  $CuSO_4$ . This gave  $p\text{-C}_6\text{H}_4(SO_3H)_2$ , which was identified as the chloride and the amide. A was said to be 3,6- $(HO_2S)_2C_6H_3NH_2$ . Since this proof of the constitution left something to be desired O. reports his own expts. The steps were the following:



$C_6H_2Cl_4$ , and Schultz's constitution for A was confirmed. 15 g. A prepd. according to Zander were dissolved in 120 cc. of concd.  $HCl$  + 40 cc.  $H_2O$ , cooled with ice and treated with excess of  $N_2O_5$ . Finally the excess of  $N_2O_5$  was removed with an air current. Gattermann's  $Cu$  was added until  $N_2$  ceased to be evolved. Then 2 mols.  $KOH$  were added for each mol. A used and the soln. was filtered, the  $HCl$  removed by evapn. and the  $Cu$  with  $H_2S$ . On filtering and evapg. dipotassium chlorodensene-2,5-disulfonate was obtained. This heated with a slight excess of  $PCl_5$  at  $140-50^\circ$  gave 60% yield of the chloride (B), m.  $96.5-97^\circ$ . 1 part B + 3 parts  $PCl_5$  heated 4 hrs. at  $210^\circ$  in the sealed tube, transferred to  $H_2O$ , extd. with  $Et_2O$  and crystd. from 80%  $EtOH$  gave  $asym\text{-}C_6H_3Cl_4$ , m.  $17-8^\circ$ . The constitution of A thus

confirmed is concordant with Holleman's rules on the introduction of substituents in the  $C_6H_4$  ring, which rules in this case exclude the  $SO_2H$  from position 5.

E. J. WITZEMANN

The dipeptide of aspartic acid and the function of asparagine in the plant. C. RAVENNA AND G. BOSINELLI. Univ. Bologna. *Gazz. chim. ital.* 50, I, 281-8(1920).—It was previously shown (*idem* 49, II, 303(1919); cf. C. A. 14, 2172, 2648) that prolonged boiling of aq. asparagine gives asparagylaspartic acid (A). Heating this at  $210^\circ$  gave an anhydride which was identical with the so-called fumaric imide and which with  $Ba(OH)_2$  gave A. Other uncertain observations were made and so the work was repeated. 10 g. asparagine in 200 cc.  $H_2O$  were boiled under a condenser and after 15-20 hrs. showed the presence of the dipeptide by giving a ppt. with neutral  $Pb(OAc)_2$ . The boiling was continued 200 hrs., after which the  $Pb(OAc)_2$  tests did not increase appreciably. The entire liquid remaining (165 cc.) was then treated with a slight excess of  $Pb(OAc)_2$ , and the ppt. washed repeatedly and finally decompd. with  $H_2S$ . The filtrate on evapn. gave 2 g. of residue, which were purified and on analysis gave the correct values for A. Other authors (Erlenmeyer, C. A. 8, 141, and Ehrlich and Lange, C. A. 8, 91) have made fruitless attempts to obtain A in this way. The mother liquors from which A was pptd. gave, on treating as described, 3 g. of crystals of aspartic acid containing a little unchanged asparagine. This is a good recovery and the results show that about half of the asparagine is converted into A and the other half into  $NH_4$  aspartate. A 5% soln. of aspartic acid boiled similarly failed to give a demonstrable trace of A and the material used was recovered unchanged. Since it might be suggested that A is formed from  $NH_4$  aspartate a 4% soln. of this compd. was boiled as before and although the soln. darkened, no A was obtained and the aspartic acid and  $NH_4$  were recovered unchanged. These results therefore indicate that asparagine may form polypeptides under conditions in which the simpler  $NH_2$  acids cannot do so, and that asparagine is a step in the regeneration of proteins. R. and B. then discuss the isomeric formulas of A obtainable from Piutti's (*Gazz. chim. ital.* 18, 457(1888)) and Fischer and Koenig's (C. A. 1, 2088(1907))  $\alpha$  and  $\beta$  formulas for asparagine. They point out that since A is amorphous it will be difficult to det. its constitution until other distinctive properties of the isomers are found.

E. J. WITZEMANN

The spontaneous decomposition of  $\alpha$ -naphthylphenylaminomethane. II. S. BERLINGOZZI. Univ. Siena. *Gazz. chim. ital.* 50, I, 321-5(1920).—In a previous paper (cf. C. A. 14, 3410) it was stated that in reducing  $\alpha$ -naphthyl phenyl ketone (A) with  $Zn + HCl$  no rearrangement occurred and that  $\alpha$ -naphthylphenylaminomethane (B) is obtained. The  $HCl$  deriv. of (B) was identical with that obtained by Busch and Leeftinck (C. A. 2, 2230) but the free base was different from that described by them. B. desired to explain this disparity. First B. noticed that the  $Et_2O$  mother liquors of the base, m.  $56-9^\circ$ , obtained in reducing A slowly evolve  $NH_3$  on standing even in pure  $Et_2O$ . At the same time a white powder is pptd. The first portion of this m.  $80-100^\circ$ . The later portions m.  $170-5^\circ$ . This made B. doubt whether the compd. m.  $121^\circ$ , described by B. and L. as B is not a corresponding partial decompn. product. The prepn. of B from  $\alpha$ - $C_{10}H_7MgBr$  and hydrobenzamide (according to B. and L.) was repeated by B. and gave the base, m.  $56-9^\circ$ , which he obtained from A and on standing the  $Et_2O$  solns. decompd. in the same way, giving the same product which when pure m.  $181^\circ$ , and is  $\alpha$ -naphthylbenzylamine.

E. J. WITZEMANN

Some derivatives of nitrotoluidine. A. G. C. LEONARD AND AGNES BROWNE. *Sci. Proc. Roy. Dublin Soc.* 16, 105-8(1920).—By the usual methods of diazotizing and coupling, the following diazo compds. have been made from 5-nitro-*o*-toluidine, 24, the latter having been prepd. according to the method of Nölting and Stoecklin (*Ber.* 566): 5-Nitro-2-, methylphenylazo- $\beta$ -naphthol (A), bright red compd. m.  $204^\circ$  (decompn.), insol. in  $H_2O$ ,  $HCl$  and  $NaOH$ ; sol. in  $C_6H_6$ ,  $Et_2O$  and  $EtOH$ ; crystals in long, red needles

from the latter solvent. Applied similarly to *p*-nitroaniline red, it dyes cotton a deep orange color. 5-Nitro-2-methylbenzenediazoamino-*o*-toluidine (B), needle-shaped crystals from alc., m. 133°, decompd. by boiling with HCl, H<sub>2</sub>SO<sub>4</sub> and AcOH, giving 6-nitro-cresol, toluidine and N. Heated with HCl and β-naphthol, 12% of the diazoamino compd. reacted to form A; the remainder decomps. as just stated. A product composed of reddish, cubical crystals, m. 144° from alc. and believed to be the isomeric 4-amino-3,2'-dimethyl-5-nitroazobenzene, was obtained by concn. of the mother liquor from the crystn. of the above compd. 5-Nitro-2-methylbenzenediazoamino-*p*-toluene (C), yellow, m. 131° (decompn.), behaved similarly to B on treatment with acids. 5-Nitro-2-methyldiazoaminobenzene-*p*-sulfonic acid (D), yellow, amorphous substance, m. 129° (decompn.), insol. in H<sub>2</sub>O, alc., Et<sub>2</sub>O and C<sub>6</sub>H<sub>6</sub> in the cold, slightly sol. on heating, decomps. on warming with acids, evolving N. 5-Nitro-2-methyldiazoamino-*p*-nitrobenzene (E), yellow, m. 118° (decompn.), sol. in warm alc., insol. in Et<sub>2</sub>O and C<sub>6</sub>H<sub>6</sub>, reacts like D on treatment with acids. 5-Nitro-2-methyl-2',4'-dihydroxyazobenzene, orange, m. 234°, fairly sol. in hot alc., sparingly sol. in C<sub>6</sub>H<sub>6</sub> and petr. ether.

A. T. FRASCATI

Some synthetic peptides. MILLE L. PETRESCU. Univ. Bucarest. *Bull. soc. chim. Romania* 1, 58-60 (1919).—Glycine derivs. were prepd., isomeric with those obtained by Schmidlin (*Ann.* 340, 123). PhCH(NH<sub>2</sub>)CN, digested with concd. HCl, gave PhCH(NH<sub>2</sub>)CO<sub>2</sub>H. To 9.5 g. in 95 cc. *N* NaOH were added, with cooling and agitation, 10 g. BrCH<sub>2</sub>COCl in 63.5 cc. *N* NaOH. The addition of 95 cc. *N* HCl gave bromoacetylphenylglycine, BrCH<sub>2</sub>CONHCHPhCO<sub>2</sub>H, white scales from hot C<sub>6</sub>H<sub>6</sub>, m. 140°, difficultly sol. in cold H<sub>2</sub>O, more easily in hot. With concd. NH<sub>4</sub>OH this gave glycylphenylglycine, NH<sub>2</sub>CH<sub>2</sub>CONHCHPhCO<sub>2</sub>H, m. 226° (decompn.); Cu salt, small blue crystals, difficultly sol. in cold H<sub>2</sub>O. Glycylphenylglycine anhydride, NH<sub>2</sub>CH<sub>2</sub>CO.NH.CHPh.CO, was made by the action of HCl gas in abs. alc., concn.

in a vacuum at 40°, and subsequent digestion with NH<sub>3</sub> in abs. alc.; octahedrons, which become brown at 230° and m. 232°. Unlike the dipeptides, the anhydride is more sol. in HCl than in H<sub>2</sub>O.

E. V. LYNN

Reactions of chloropicrin. GUILLEMAND and LABAT. *Bull. soc. pharm. Bordeaux* 1919; *Ann. chim. anal.* 2, 120-1 (1920).—Alkali polysulfide soln. destroys the odor of chloropicrin. A yellow color is obtained when a drop of chloropicrin is boiled with alc. KOH and a small quant. of thymol. The substitution of resorcinol for thymol produces a red color. The addition of H<sub>2</sub>SO<sub>4</sub> to the thymol mixt. produces a reddish violet color, and the whole mixt. on diln. with AcOH exhibits an absorption band in the green portion of the spectrum. A mixt. of CHCl<sub>3</sub> and a trace of NaNO<sub>2</sub> gives the same reactions. Both CHCl<sub>3</sub> and chloropicrin yield a carbylamine. Chloropicrin, when boiled with KOH soln., gives, after cooling, reactions characteristic of HNO<sub>2</sub>.

A. G. DuMaz

Beckmann rearrangement. IX. Action of phosphorus pentasulfide upon benzo-phenoxime. MITSURU KUHARA AND KÔZÔ KASHIMA. *Mem. Coll. Sci. Kyoto Imp. Univ.* 4, 69-75 (1919).—Dodge (*Ann.* 264, 184 (1891)) and Ciusa (*Atti acad. Lincei*, 15, II, 379-84 (1906) I, 28) obtained PhCSNHPh by the action of P<sub>2</sub>O<sub>5</sub> upon PhC:NOH, using C<sub>6</sub>H<sub>6</sub> and CS<sub>2</sub> as solvents, resp., without isolating any intermediate substance. The scheme of the reaction would be as follows: PhC:NOH → PhC:NSH → PhC:(NPh)SH ⇌ PhC(NHPh)S. Considering the reaction to be due to the rearrangement of a phosphoryl ester of Ph<sub>2</sub>CS, which possibly formed as an intermediate product, K. and K. conducted the reaction, using 5 mols. of PhC:NOH, 1 mol. of P<sub>2</sub>S<sub>5</sub> and 1.5 mols. of P<sub>2</sub>O<sub>5</sub> as dehydrating agent and Et<sub>2</sub>O as solvent. On expelling the solvent, an intermediate substance, thiobenzophenoxime oxyphosphoryl ester, (Ph<sub>2</sub>C:NS)<sub>2</sub>PO.OH, was obtained in greenish yellow fibers. It is very unstable and suffers rearrangement with

an explosive violence by mere heating to 70°, yielding  $\text{PhCSNHPH}$  by suitable treatment. Treated with alc. KOH at room temp., *thiobenzophenoxime hydrogen dipotassium orthophosphate*,  $(\text{Ph}_2\text{C:NS})_2\text{P(OK)}_2\text{OH}$ , was obtained in colorless needles, rearranges at 149° with violence. The latter is changeable to the former by alc. HCl. K. and K. have endeavored to isolate  $\text{Ph}_2\text{C:NSH}$ , but have not succeeded. K. K.

**Organic compounds of arsenic.** I. KAORU MATSUMIYA. *Mem. Coll. Sci. Kyoto Imp. Univ.* 4, 217-23 (1920).—By the action of halides of metals or metalloids upon the Barbier-Grignard reagent, Pfeiffer obtained many organo-metallic or metalloid compds. According to the method, M. studied the action of  $\text{AsCl}_3$  upon  $\text{PhMgBr}$  (A) and  $\alpha\text{-C}_{10}\text{H}_7\text{-MgBr}$  (B), obtaining mainly  $\text{AsPh}_3$ , m. 59-60°, and  $\text{As(C}_{10}\text{H}_7)_3$ , m. 348°. The yield of the latter was 70%, while that of the Na method was only 20%. The formation of the primary and sec. compds. was very small in each case. M., therefore, conducted the reaction in the presence of a large excess of  $\text{AsCl}_3$ , i. e., A and B are gradually added to  $\text{AsCl}_3$  in  $\text{Et}_2\text{O}$ . The products were mainly in these cases 12 g. of  $\text{Ph}_3\text{AsCl}$ , m. 173-4°, from 32.5 g. of  $\text{PhBr}$ , and 21 g. of *di- $\alpha$ -naphthylarsenious chloride* from 41.4 g. of  $\alpha\text{-C}_{10}\text{H}_7\text{Br}$ , i. e., in 60% yield. The latter formed slightly yellow, fine crystals, m. 166-7°. K. K.

**Ditertiary hydrazines. XXII. Basic tetraarylhydrazines.** HEINRICH WIELAND. *Akad. Wiss. und Techn. Hochschule, München. Ber.* 53B, 1313-28 (1920); cf. C. A. 13, 2873.—The continuation of the study of the ditertiary aromatic hydrazines has fully confirmed the principle that dissociation at the hydrazine union into free radicals with bivalent N is increased by positive substituents on the  $\text{C}_6\text{H}_4$  nuclei. If it is attempted to apply the otherwise so useful method of oxidation of diarylamines to the synthesis of the simplest basic hydrazine,  $[\text{Ph(p-H}_2\text{NC}_6\text{H}_4\text{N)}_2]$ , the competing change into quinone derivs. comes into play: *p*- $\text{H}_2\text{NC}_6\text{H}_4\text{NHPH}$  oxidized as the free base gives  $\text{HN:C}_6\text{H}_4\text{:NHPH}$ , which in acid soln. polymerizes to emeraldine, but when 5 g. *p*- $\text{AcNH-C}_6\text{H}_4\text{NHPH}$  in 60 cc. cold  $\text{Me}_2\text{CO}$  "stabilized" with  $\text{KMnO}_4$  is treated with 4 g. very finely powdered  $\text{KMnO}_4$  in small portions, *p,p'*-*di*[*acetylamino*]tetraphenylhydrazine is obtained in prisms from alc., begins to decomp. 100°, m. 124°, is somewhat sensitive to light, gives with glacial  $\text{AcOH}$  or in  $\text{Me}_2\text{CO}$  with HCl in  $\text{Et}_2\text{O}$  a violet color quickly changing to green; thus far it has not been possible to hydrolyze off the Ac groups, both acids and alkalis decomp. the substance. *p*-Diethylaminodiphenylamine (12 g. from 21 g. *p*- $\text{ONC}_6\text{H}_4\text{NEt}_2$  in 200 cc. alc. and 100 cc.  $\text{Et}_2\text{O}$  treated with 15.5 g.  $\text{PhNH-NH}_2$ , freed from the  $\text{Et}_2\text{O}$  by distn., dild. with 75 cc.  $\text{H}_2\text{O}$  and allowed to stand overnight), leaves from ligroin, m. 88-9°; 1.5 g. in 50 cc. cold  $\text{Et}_2\text{O}$  shaken with 1.8 g. dry  $\text{Ag}_2\text{O}$  added in 0.3-0.5 g. portions at 15-min. intervals, filtered after 1 hr., extd. from the Ag with  $\text{C}_6\text{H}_6$  and pptd. with petr. ether, gives *p,p'*-tetraethyldiaminotetraphenylhydrazine, felted crystals, m. 104°, sol. in  $\text{C}_6\text{H}_6$  with greenish yellow color changing to olive-green on rapid warming and back to almost the original shade on immediate cooling, a process which can be repeated 3-4 times. It dissolves in  $\text{AcOH}$  without color, becoming blue only on long boiling. Shaken in  $\text{Et}_2\text{O}$  24 hrs. with  $\text{H}_2\text{O}$  it gives  $\text{Et}_2\text{NC}_6\text{H}_4\text{NHPH}$ ,  $\text{O:C}_6\text{H}_4\text{:NPh}$  and  $\text{Et}_2\text{NH}$ . With NO in  $\text{C}_6\text{H}_6\text{-Et}_2\text{O}$ , it yields a *nitrosamine*,  $\text{Et}_2\text{NC}_6\text{H}_4\text{N(NO)Ph}$ , yellow needles from petr. ether, m. 78°, while 1.5 g. allowed to stand overnight with 2 g. freshly prepd.  $\text{C}_2\text{Ph}_4$  in 10 cc.  $\text{C}_6\text{H}_6$  in  $\text{CO}_2$  and then slowly pptd. with alc. yields a compound  $\text{Et}_2\text{NC}_6\text{H}_4\text{NPhCPh}_3$ , stout prisms from  $\text{C}_6\text{H}_6\text{-EtOH}$ , m. 163°. *p*-Methoxy-*p'*-dimethylaminodiphenylamine (A), obtained in 35-40% yield by boiling 25 g. *p*- $\text{IC}_6\text{H}_4\text{OMe}$ , 17 g. *p*- $\text{H}_2\text{NC}_6\text{H}_4\text{NHAc}$ , 8 g.  $\text{K}_2\text{CO}_3$ , 0.3 g. Cu bronze and 0.1 g. I 18 hrs. in 125 cc.  $\text{PhNO}_2$ , removing the  $\text{PhNO}_2$  with steam, taking up in  $\text{Et}_2\text{O}$  and alc., evapg., boiling 3 hrs. with 100 cc. concd. HCl and 25 g.  $\text{SnCl}_4$ , cooling, treating with  $\text{Et}_2\text{O}$  and  $\text{H}_2\text{O}$  and decomp. the cryst.  $\text{SnCl}_4$  salt which seps. after a time with excess of cold NaOH, leaflets from ligroin, m. 78°; it can also be purified, without passing through the  $\text{SnCl}_4$  salt, by hydrolyzing in the presence of only

3-5 g.  $\text{SnCl}_4$  (to protect it from autoxidation), pouring into excess of alkali, extg. with  $\text{Et}_2\text{O}$  and distg. under 1 mm. at about  $200^\circ$ . Oxidized in  $\text{C}_6\text{H}_5\text{N-Et}_2\text{O}$  with  $\text{Ag}_2\text{O}$  as described for  $(\text{Me}_2\text{NC}_6\text{H}_4)_2\text{NH}$  (B) (C. A. 9, 2537) it gives *p*-dimethoxytetramethyldiaminotetraphenylhydrazine (yield of crude product, 70-80%); it is purified by sepg. it mechanically from the excess of  $\text{Ag}_2\text{O}$  in petr. ether suspension, washing with cold  $\text{Et}_2\text{O}$ , dissolving as quickly as possible at room temp. in 2-3 cc.  $\text{CHCl}_3$ , adding 5 cc. dry  $\text{Et}_2\text{O}$  after 1-2 min. and filtering into a flask in a freezing mixt. On rubbing, it seps. in quadratic prisms, m.  $93^\circ$ , decompd. by alcs. and  $\text{H}_2\text{O}$ , sol. in cold  $\text{C}_6\text{H}_6$  with pure yellow color, becoming olive at about  $60^\circ$  and again yellow on rapid cooling. With  $\text{NO}$  it gives quickly in  $\text{C}_6\text{H}_6$  the nitrosamine of A, deep yellow leaflets from alc., m.  $95^\circ$ , also obtained from A and  $\text{AmONO}$ . From 15 g. *p*- $\text{ONC}_6\text{H}_4\text{NEt}_2$  reduced to decolorization in 60 cc.  $\text{HCl}$  and 200 cc.  $\text{H}_2\text{O}$  with  $\text{Zn}$  dust, filtered, treated with 12.4 g.  $\text{PhNEt}_2$ , then with 20 cc. concd.  $\text{HCl}$ , poured with cooling and turbing into a cold satd. soln. of 8.1 g.  $\text{K}_2\text{Cr}_2\text{O}_7$  and rubbed is obtained 12-5 g. of the ethylindamine-zinc chloride compound, crystals with red luster, a warm soln. of 5 g. of which slowly poured into 4 g.  $\text{Na}_2\text{S}_2\text{O}_4$ , 20 cc. of 20%  $\text{KOH}$  and 10 cc.  $\text{NH}_4\text{OH}$  in 200 cc. of  $\text{H}_2\text{O}$  gives *p,p'*-tetraethyl-diaminodiphenylamine, crystals from ligroin, m.  $56^\circ$ ; whereas the tetra-Me compd. (B) can be oxidized to the hydrazine, the Et compd. shaken 1 hr. in ligroin with  $\text{Ag}_2\text{O}$  gives diethylindophenol, crystals with green luster from ligroin, m.  $106^\circ$ . Evidently the hydrazine is formed, but dissociates into the radicals  $(\text{Et}_2\text{NC}_6\text{H}_4)_2\text{N}$ , which are decompd. by the  $\text{H}_2\text{O}$  produced by the oxidation into  $\text{Et}_2\text{NC}_6\text{H}_4\text{N}:\text{C}_6\text{H}_4:\text{O} + (\text{Et}_2\text{NC}_6\text{H}_4)_2\text{NH}$ . Similarly,  $[2,4,6-(\text{MeO})_3\text{C}_6\text{H}_2]_2\text{NH}$  shaken with  $\text{Ag}_2\text{O}$  in  $\text{Me}_2\text{CO}$  quickly forms a deep violet soln. from which, after filtering and distg. off most of the  $\text{Me}_2\text{CO}$ , pentamethoxyquinone anil seps. in black-green crystals rubbing to a violet powder, m.  $202^\circ$ , insol. in alkalis, decompd. by hot acids, sol. in  $\text{C}_6\text{H}_6$  with almost cherry-red, in  $\text{Me}_2\text{CO}$  with red-violet, in alc. and in concd.  $\text{H}_2\text{SO}_4$  with blue color, sublimes in purple vapors, reduced in alc. by  $\text{Zn}$  dust and  $\text{AcOH}$  on gentle warming to the leuco compound, needles from dil. alc., decomp.  $156-7^\circ$ , sol. in alkalis but quickly converted by atm. O into the insol. quinone anil. Oxidation of B gives  $\text{Me}_2\text{NC}_6\text{H}_4\text{N}:\text{C}_6\text{H}_4:\text{NMe}$  (C) besides the hydrazine; the earlier assumption that this was due to elimination of H and Me from B by the  $\text{Ag}_2\text{O}$  was erroneous, for it has now been found that an  $\text{Et}_2\text{O}$  soln. of B oxidized to the hydrazine stage gives only little C, but that the amt. of the latter increases markedly if the soln. is subjected to the further action of an excess of  $\text{Ag}_2\text{O}$ ; the C is, therefore, formed from the radical  $(\text{Me}_2\text{NC}_6\text{H}_4)_2\text{N}$ ; the change is effected not only by oxidizing agents but also by illuminating or heating solns. of the radical. Thus, if 4 g. B in 60 cc. cold  $\text{Et}_2\text{O}$  is treated with 3 g.  $\text{Ag}_2\text{O}$  in 3 portions at 0.5-hr. intervals and shaken 3.5 hrs.; a portion of the soln. with  $\text{NO}$  gives  $(\text{Me}_2\text{NC}_6\text{H}_4)_2\text{NNO}$ , indicating the presence of the radical. The rest of the soln. (35 cc.), shaken 4 hrs. more with 5 g.  $\text{Ag}_2\text{O}$ , and treated with  $\text{NO}$ , gives the bisnitrosoamine,  $\text{Me}_2\text{NC}_6\text{H}_4\text{N}(\text{NO})\text{C}_6\text{H}_4\text{N}(\text{NO})\text{Me}$ , formed by addition to C; this compd. is obtained in 4-g. yield by shaking 8 g. B in 150 cc.  $\text{Et}_2\text{O}$  4 hrs. with 28 g.  $\text{Ag}_2\text{O}$ , filtering and satg. with  $\text{NO}$ ; it seps. from alc. in yellow-green crystals, sinters  $130^\circ$ , m.  $136^\circ$ , forms brownish green hot satd. solns., loses  $\text{NO}$  with formation of C in boiling xylene, reduced by  $\text{SnCl}_2$  and  $\text{HCl}$  to *p,p'*-trimethyldiaminodiphenylmethane, needles from gasoline, m.  $73^\circ$ , which, shaken 10-5 min. in  $\text{Et}_2\text{O}$  with  $\text{Ag}_2\text{O}$ , yields trimethylindamine (C), leaflets with faint bronze luster, m.  $90^\circ$ , forms cherry-red solns. (those of its salts are green), smoothly adds  $\text{NO}$  to give the above bisnitrosoamine, slowly adds  $\text{Ph}_3\text{C}$ . XXIII. Mixed aromatic ditertiary hydrazine and tetrabenzyldiazine. HEINRICH WIELAND AND EDUARD SCHAMBERG. *Ibid* 1329-35.—Substitution of alkyl for two of the aryl groups in ditertiary aromatic hydrazines diminishes the ability to dissociate at the N.N union (C. A. 7, 598); this is confirmed in the present paper by a study of *N,N*-dimethyl-*N',N'*-diphenylhydrazine (A), which is unchanged by boiling for hrs. in xylene; the introduction of the two Me groups, how-



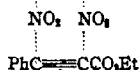
ever, does produce some weakening of the N.N union as compared with that in  $\text{Ph}_2\text{NNH}_2$ , for while the latter loses the  $\text{NH}_2$  group as  $\text{NH}_3$  only with concd.  $\text{H}_2\text{SO}_4$  (C/A. 8, 346), A with dil. aq. HCl first forms the normal salt, which, however, soon decomps., even in the cold, into  $\text{NHMe}_2$ ,  $\text{NHPh}_2$  and complex dyestuffs. A, prisms, m.  $52^\circ$ , is obtained by mixing 10 g. freshly distilled  $\text{Ph}_2\text{NNH}_2$  with 8 g.  $\text{Me}_3\text{SO}_4$  in the cold, treating with 1.3 g. Na after the reaction is over, then again, without cooling, with 9 g.  $\text{Me}_3\text{SO}_4$ , allowing to stand some hrs., making alk. with NaOH, pouring into  $\text{H}_2\text{O}$ , extg. with  $\text{Et}_2\text{O}$ , shaking repeatedly with  $\text{H}_2\text{O}$  to remove the alc., drying with  $\text{K}_2\text{CO}_3$  and fractionally pptg. with HCl in  $\text{Et}_2\text{O}$ , the HCl salt of unchanged  $\text{Ph}_2\text{NNH}_2$  sepg. first. When 1 g. is dissolved in 20 cc. of 15% HCl the soln. quickly changes through yellow to red and on the following day to dirty green and deposits a dark green dye; the soln. contains 30% of the theoretical amt. of  $\text{NHMe}_2$  and  $\text{Et}_2\text{O}$  exts. from the dye 0.42 g.  $\text{Ph}_2\text{NH}$ . The dye (1.8 g. from 4.6 g. A) with Zn dust and AcOH gives the *leuco compound*,  $\text{C}_{20}\text{H}_{21}\text{N}_3$ , crystals from petr. ether, m.  $175-6^\circ$ . No method has yet been discovered for the prepn. of aliphatic ditertiary hydrazines.  $(\text{MeNH})_2\cdot 2\text{HCl}$  with NaOH and MeI in MeOH gives *N-methyl-N'-trimethylhydrazonium iodide*, prisms from  $\text{Me}_2\text{CO}$ , m.  $226.5^\circ$ .  $(\text{PhCH}_2)_2\text{NNHCH}_2\text{Ph}$ , however, heated 0.5 hr. on the  $\text{H}_2\text{O}$  bath with 0.5 part  $\text{PhCH}_2\text{Br}$ , and treated with  $\text{Et}_2\text{O}$  to ppt. the  $(\text{PhCH}_2)_2\text{NNHCH}_2\text{Ph}$  gives almost quant. *tetrahensylhydrazine*, prisms from alc., m.  $139.5^\circ$ , forms no salts, is unchanged by boiling 32 hrs. in 20 cc. alc. with 1 g. Zn dust and 5 cc. AcOH. Heated at  $260^\circ$  under 32 mm., it partly dists. without decompn. and partly decomps. into  $(\text{PhCH}_2)_2\text{NH}$  and  $\text{PhCH}_2\text{N}:\text{CHPh}$ . It dissolves without change in concd.  $\text{H}_2\text{SO}_4$ , but when heated 2 hrs. at  $160^\circ$  and 1 hr. at  $205^\circ$  with concd. HCl it decomps. into  $\text{N}_2\text{H}_4$  and  $\text{PhCH}_2\text{HCl}$ . Attempts to oxidize dicyclohexylamine (8 g.) to the hydrazine by gradually treating it in 200 cc.  $\text{Me}_2\text{CO}$  with  $\text{KMnO}_4$  and shaking until the color persisted for 1 hr., yielded 4 g. *adipic cyclohexylamide*, needles from  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_4$  or alc., m.  $138-9^\circ$ , hydrolyzed by heating 6 hrs. at  $150^\circ$  with 20% HCl to adipic acid and  $\text{C}_6\text{H}_{11}\text{NH}_2$ .

**XXIV. Ditary hydrazines of the quinoline series.** HEINRICH WIELAND and EMMY HAAS. *Ibid* 1336-43.—The tendency to dissociation of the following hydrazines of the tetrahydroquinoline series is greatly diminished as compared with the dialkyl-diarylhydrazines. No color appears on boiling in xylene and only in high-boiling solvents (as  $\text{BzOEt}$ ) is a yellow to brown color produced, an indication of dissociation into radicals of bivalent N, which, under the conditions, undergo further change, one product of which is the sec. amine, tetrahydroquinoline. These "quinolinehydrazines" have no basic properties; they are insol. in dil. mineral acids and form no salts with HCl in  $\text{Et}_2\text{O}$ , but they are changed by acids more or less rapidly, depending on the conditions. *N,N'-Bis-1,2,3,4-tetrahydroquinolyl* (A), obtained in 40% yield from 50 g. tetrahydroquinoline in 900 cc. cold stabilized  $\text{Me}_2\text{CO}$  slowly treated with  $\text{KMnO}_4$  until the color persists for some time, yellowish prisms from  $\text{C}_6\text{H}_5\text{-EtOH}$ , m.  $141-2^\circ$ , sol. in concd.  $\text{H}_2\text{SO}_4$  with brown-red color changing to blood-red with a few drops of  $\text{H}_2\text{O}$ , gives with HCl in  $\text{Et}_2\text{O}$  an amorphous brown-yellow decompn. product; glacial AcOH seems to produce a similar cleavage; these colored products, which probably belong to the perazine series, are formed, together with tetrahydroquinoline, by cleavage at the N. Dil. (2 N) HCl effects this cleavage so slowly that the latter reaction is almost completely supplanted by a benzidine rearrangement; 20 g. A in satd.  $\text{Et}_2\text{O}$  soln. shaken 12 hrs. with 300 cc. of 2 N aq. HCl yields 22 g. of a cryst. *hydrochloride of tetrahydroquinobenzidine*, needles from alc. or  $\text{Me}_2\text{CO}$ , m.  $128-9^\circ$ , is pronouncedly basic, oxidized in acid soln. by  $\text{FeCl}_3$  or  $\text{CrO}_3$  to a yellow-red quinoid salt, sol. with a similar color in concd.  $\text{H}_2\text{SO}_4$ . *N,N'-Dinitrosamine*, broad golden yellow rods from  $\text{C}_6\text{H}_4$ , m.  $196^\circ$ , sol. in  $\text{H}_2\text{SO}_4$  with yellow-red color, loses NO in boiling  $\text{BzOEt}$ . *N,N'-Bis-p-tolyltetrahydroquinolyl* (from p-tolyltetrahydroquinoline; yield, 40-50%), needles from alc., m.  $107-8^\circ$ , decompd. by acids (including 2 N HCl) into the quinoline and colored products, sol. in AcOH with green

color changing through olive to yellow-brown, in  $\text{H}_2\text{SO}_4$  with dirty violet color changing through blue-violet and lilac to red-brown. *N,N'*-Bis-*p*-methoxytetrahydroquinolyl (yield, 40%), crystals from  $\text{C}_6\text{H}_5\text{-EtOH}$  or from  $\text{Me}_2\text{CO}$ , m.  $117-8^\circ$ , sol. to the extent of 0.3% in  $\text{Et}_2\text{O}$ , more easily decompd. by acids than the preceding compds., sol. in glacial  $\text{AcOH}$  with pink, in concd.  $\text{HCl}$  with dark red, in 2 *N*  $\text{HCl}$  with emerald-green, in concd.  $\text{H}_2\text{SO}_4$  with brown-violet color, changed by  $\text{H}_2\text{O}$  to green. When shaken 5-6 hrs. in  $\text{Et}_2\text{O}$  with 14 parts of 2 *N*  $\text{HCl}$  and, when the reaction is over (the emerald-green soln. changes to reddish olive-brown), poured into a soln. of the amt. of  $\text{NaOH}$  necessary for neutralization and containing 0.25 as much  $\text{Na}_2\text{S}_2\text{O}_4$  as the original hydrazine, it yields 16-8% of the *perazine*,  $\text{C}_{20}\text{H}_{22}\text{O}_4\text{N}_4$ , yellow crystals from  $\text{AcOEt}$ , m.  $160^\circ$ , mol. wt. in freezing  $\text{C}_6\text{H}_6$  313; the  $\text{Et}_2\text{O}$  soln. with  $\text{HCl}$  soon turns red and deposits the scarlet-red needles of the *meri*-quinoid azonium chloride formed by autoxidation. The *perazine* does not dissolve in dil. aq.  $\text{HCl}$ ; with  $\text{H}_2\text{SO}_4$  it first gives a cherry-red soln., which then shows red-olive fluorescence;  $\text{H}_2\text{O}$  changes it through violet to pink.

CHAS. A. ROUILLER.

Addition of nitrogen dioxide to phenylpropionic ester. HEINRICH WIELAND with HANS WAGNER. Techn. Hochschule, München. Ber. 53B, 1343-6(1920).—When 2 g.  $\text{PhC:CCO}_2\text{Et}$  in 5 cc. dry gasoline (b.  $40-50^\circ$ ) in a freezing mixt. is treated with a slow current of pure  $\text{NO}_2$  (dried over  $\text{P}_2\text{O}_5$  and  $\text{PhO}$ ) until it increases 0.9 g. in wt. an addition product (A) seps. in prisms. If attempts are made to filter it off it soon begins to deliquesce with evolution of  $\text{NO}_2$ ; on clay it quickly disappears; it easily dissolves in  $\text{Et}_2\text{O}$  without color. Indirect analyses made by shaking 0.2-0.3 g. in 40 cc. 0.1 *N*  $\text{KOH}$  under  $\text{Et}_2\text{O}$  and detg. the  $\text{PhC:CCO}_2\text{Et}$  in the  $\text{Et}_2\text{O}$  by weighing the residue and the  $\text{NO}_2$  in the alkali (as  $\text{KNO}_3 + \text{KNO}_2$ ) by titrating with  $\text{HCl}$  and phenolphthalein showed that the components are present in A in the ratio 1:1. If the treatment of the  $\text{PhC:CCO}_2\text{Et}$  with  $\text{NO}_2$  is continued until 2 mols.  $\text{NO}_2$  have been absorbed, the stable addition product, ethyl  $\alpha,\beta$ -dinitrocinnamate (B), is obtained as a yellow oil which on short standing deposits almost colorless crystals, m.  $66.5^\circ$ . If A is allowed to stand in a closed vessel, it decomp. into equal parts of B and  $\text{PhC:CCO}_2\text{Et}$ . W. believes A is not a normal but a subsidiary valence compd.  $\text{PhC}\equiv\text{CCO}_2\text{Et}$



CHAS. A. ROUILLER

Benzene problem. III. A. v. WEINBERG. Ber. 53B, 1353-8(1920); cf. C. A. 14, 1332.—Debye and Scherrer's hypothesis (C. A. 12, 786) that C in diamond form may be considered the prototype of aliphatic, graphite and amorphous<sup>c</sup> that of aromatic compds. does not seem to be established from the chemical standpoint. The lattices of both the diamond and graphite contain exclusively 6-membered rings, hydroaromatic cyclohexane rings. The fact that mellitic acid can be obtained from graphite and not from the diamond is no chemical proof of the aromatic nature of graphite. Moreover, graphite is attacked only with the greatest difficulty and yields only graphitic acid at first; that the diamond is not attacked proves nothing as to its aliphatic nature; if it were attacked there is theoretically no reason why its cyclohexane rings should not yield mellitic acid just as well as those of graphite, for even with graphite as the starting point the C-ring is no longer unchanged; the 6-membered ring in mellitic acid (i. e., the  $\text{C}_6\text{H}_6$  ring) is more than 4 times as extensive as that in graphite ( $1.45 \times 10^{-8}$ :  $6.2 \times 10^{-8}$ ). v. Steiger has recently (C. A. 14, 3232) attempted to support D. and S.'s theory by means of the heats of combustion of aromatic compds. but as a matter of fact the heat of combustion of  $\text{C}_6\text{H}_6$  speaks against the theory. Assuming that the work of dissociating C-H unions in  $\text{C}_6\text{H}_6$  is the same as in satd. aliphatic hydro-

carbons, the portion of the heat of combustion due to the 6 H atoms (6y) is 180 Cal. Deducting this from the heat of combustion of  $C_6H_6$  vapor, 787.7 Cal., there remains 607.7 Cal., or the value ( $x_B$ ) for the setting free and combustion of each C atom is 101.3 Cal. as against  $x = 96$  for satd. aliphatic compds. The difference (5.3 Cal.) has also been found for other aromatic hydrocarbons. For graphite  $x = 94.3$ , a difference of 7, or 42 Cal. for each 6-membered ring. v. W. does not agree with Fajans (C. A. 14, 3086) that "in the formation of a double or triple union from the atoms there is not a storing but an evolution of energy." Assuming that  $Q_{CH}$  is the same in  $C_6H_6$ ,  $C_6H_4$ , and  $C_6H_2$ , at  $18^\circ$   $x$  for each C atom in the 3 compds. is 95.5, 110 and 127.4 Cal., i. e., the doubly bound has 14.5 and the triply bound 31.9 Cal. more energy than the singly bound C atom.

CHAS. A. ROULLER

**Pyrocatecholarsenic acids.** II. R. F. WEINLAND AND J. HEINZLER. Univ. Tübingen. Ber. 53B, 1358-68(1920); cf. C. A. 14, 63.—The tetrahydrate of the tripyrocatecholarsenic acid (A),  $[OAs(OC_6H_4O)_3]H_2$ , described in the earlier paper forms rhombic bipyramidal crystals,  $a:b:c = 0.958:1:0.988$ . (100), (010), (001), (110), (111),  $T$  1.610,  $\beta$  1.582,  $\alpha$  1.563 for Na light; 100 g. of the aq. satd. soln. contains 42.4 g., of the alc. soln. 82.2 g. It hydrolyzes in  $H_2O$ , slightly in concd., completely in 1% soln. Hydrolysis is shown by the immediate green color given with  $FeCl_3$  and by the red  $Ag_3AsO_4$  instead of the white salt of A pptd. by  $AgNO_3$ . At equil. (detd. by pptg. with  $AgNO_3$ , adding just enough dil.  $HNO_3$  to dissolve the  $Ag_3AsO_4$  and weighing the Ag salt of A) at room temp., a 0.1 M soln. in  $H_2O$  contains 35.2-36.2%  $A \cdot 4H_2O$ . A dihydrate, columns, is obtained by adding concd. HCl to a concd. soln. of the tetrahydrate or by cryst. the latter from glacial AcOH, whereas on crystn. from MeOH it gives the dialcoholate  $A \cdot 2MeOH$ , columns; ethyl alcoholate,  $A \cdot 2EtOH$ , long flat prisms. The following salts are described ( $R = [OAs(OC_6H_4O)_3]$ ): Lithium,  $RH_2Li \cdot 4H_2O$ , thick 4-sided plates and squares; mercurous,  $RHHg_2$ , elongated prisms, dark red-brown when dried over  $H_2SO_4$ , absorbs about 1%  $H_2O$  in the air and becomes yellow-brown; thallium,  $RH_2Tl$ ; chromic,  $R_2H_2Cr \cdot 12H_2O$ , cubical crystals; aluminium, cubical crystals; cerium,  $R_2H_2Ce \cdot 15H_2O$ , needles; lanthanum, needles; yttrium, needles; hexamminecobaltic,  $R_2H_2[Co(NH_3)_6]$ , 4-sided yellow-brown leaflets; aquopentamminecobaltic, 4-sided brown-red leaflets; chloropentamminecobaltic, Cu-red columns; aquopentamminenickel,  $R_2H_2[Ni(NH_3)_5(H_2O)]$ , lilac tablets; monopyridine,  $RH_2 \cdot C_5H_5N$ , from 1.8 g.  $C_5H_5N$  in 100 cc.  $H_2O$  and 5 g. A in the same amt. of  $H_2O$ , or directly from 2.7 g.  $H_2As_2O_7$  and 6.6 g.  $o\text{-}C_6H_4(OH)_2$  in 40 cc. hot  $H_2O$  and 4.8 g.  $C_5H_5N$ , cryst. powder of microrodlets; dipyridine, from 3 g. A in 10-5 cc. hot  $C_5H_5N$ , thick elongated prisms continuously evolving  $C_5H_5N$  and becoming turbid; monoquinoline, from 1 g.  $C_5H_7N$  in 250 cc.  $H_2O$  containing  $H_2SO_4$  and 10 cc. of a concd. soln. of A, yellowish white powder; salt with 1.5 mols. quinoline, from 5 g. A in about 15 cc. hot  $C_5H_7N$ , long pointed canary-yellow columns; monoaniline, from 2.5 g. A and 1 cc.  $PhNH_2$  in 10 cc. hot 96% alc., columns and pyramids; dianiline, from 2 g. anhydrous A in 5-10 cc. hot  $PhNH_2$ , thick rectangular tables or columns; guanidine, crystals which under the microscope are similar to those of  $MgNH_4PO_4$ . The  $C_5H_5N$  salt is somewhat too sol. to make it possible to det.  $C_5H_5N$  gravimetrically by means of it, but  $C_5H_7N$  can be so detd. if an empirical factor is used (details will be published elsewhere). Alkaloids (morphine, quinine, strychnine, colchicine, hydrastinine, veratrine, conine, apomorphine) give ppts. with A, some at very great dilns.; so also do dil. peptone and albumin solns.

C. A. R.

**Synthesis of some  $\beta$ -amino acids from the malonate of amines and formaldehyde.** C. MANNICH AND B. KATHER. Frankfurt a/M. Ber. 53B, 1368-71(1920).—From 7.1 g.  $MeCH(CO_2H)_2$  in ice neutralized with 33%  $NHMe_3$ , treated with 7.1 g. more  $MeCH(CO_2H)_2$ , then with 12 g. of 35%  $HCHO$  and allowed to stand some time in the ice is obtained 7 g.  $\omega$ -dimethylaminodimethylmalonic acid,  $Me_2NCH_2CMe(CO_2H)_2$ , prisms from dil. alc., m. about  $98^\circ$  (foaming), forms strongly acid aq. solns., evolves no

basic vapors when boiled with NaOH; cautiously heated above its m. p., it decomps. into  $\text{CO}_2$ ,  $\text{NHMe}_2$  and  $\text{CH}_2\text{:CMeCO}_2\text{H}$ . *Methyliminobis(dimethylmalonic acid)*,  $\text{Me}_2\text{N}[\text{CH}_2\text{CMe}(\text{CO}_2\text{H})_2]_2$ , (20 g. from 11.8 g.  $\text{MeCH}(\text{CO}_2\text{H})_2$ , exactly neutralized with 33%  $\text{NH}_2\text{Me}$ , then treated with 35.4 g. more  $\text{MeCH}(\text{CO}_2\text{H})_2$ , 15 cc.  $\text{H}_2\text{O}$  and 35 g. of 35%  $\text{HCHO}$ ), pptd. by  $\text{HCl}$  from  $\text{KOH}$  in crystals, decomps.  $83-4^\circ$ , begins to deliquesce after several weeks and evolves the odor of  $\text{CH}_2\text{:CMeCO}_2\text{H}$ , quickly decomps. above its m. p. with evolution of acid vapors; if the temp. is gradually raised to  $210^\circ$ , almost all dists. as  $\text{CH}_2\text{:CMeCO}_2\text{H}$  and its  $\text{MeNH}_2$  salt,  $\beta, \beta'$ -*Tetramethyldiaminoisobutyric acid dihydrochloride* (10 g. from 10.1 g.  $\text{CH}_2(\text{CO}_2\text{H})_2$  and 27 g. of 33%  $\text{MeNH}_2$  slowly treated with 20 g. of 35%  $\text{HCHO}$ , evapd. after 24 hrs. (lively evolution of  $\text{CO}_2$  takes place), treated with 20 cc. concd.  $\text{HCl}$  and evapd. to crystn.), fine needles from dil. alc., m.  $189^\circ$ , forms a difficultly sol. double salt with  $\text{HgCl}_2$ ; free acid, fine needles from alc., m.  $139^\circ$ .  $\text{HCHO}$  (2 mols.) with 1 mol.  $\text{CH}_2(\text{CO}_2\text{H})_2$  and 1 mol.  $\text{MeNH}_2$  in  $\text{H}_2\text{O}$  condenses readily, but no satisfactory method of working up the apparently complicated product has been found; only a small amt. of a chloroaurate,  $\text{C}_8\text{H}_{10}\text{O}_4\text{N}_2\text{HAuCl}_4$ , has been isolated.

CHAS. A. ROUILLER

Occurrence of optical antipodes in nature. HANS PRINGSHEIM. *Ber.* 53B, 1372-4 (1920).—The speculations of Hess and Weltzien (*C. A.* 14, 2360) contain nothing new, but go too far; biological laws are only very rarely without exceptions. C. A. R.

Ability of plants to synthesize optical antipodes. KURT HESS. *Techn. Hochschule, Karlsruhe i. B.* *Ber.* 53B, 1375-8 (1920).—Answer to Pringsheim (preceding abstr.).

CHAS. A. ROUILLER

Preparation of 1,4-dimethylfluorenone and an attempt to convert it into a phenanthrone derivative. ALFRED SCHAARSCHMIDT AND JOHANN HERZENBERG. *Techn. Hochschule, Berlin.* *Ber.* 53B, 1388-98 (1920); cf. *C. A.* 11, 2797.—The study of the behavior of substituted fluorenones on fusion with alkali has been extended to 1,4-dimethylfluorenone (A). From the data now available, it is concluded that whereas in chrysoketone and allochrysoketone cleavage occurs in both of the two possible directions in *o*-substituted ring ketones only one  $\text{CO}_2\text{H}$  acid is formed; if the substituent is an acid group, like  $\text{CO}_2\text{H}$ , the new  $\text{CO}_2\text{H}$  group formed by the cleavage goes on the non-substituted C<sub>10</sub> ring, while if the substituent is basic ( $\text{OH}$ ,  $\text{NH}_2$ ) or a Me group, as in A, the new  $\text{CO}_2\text{H}$  group goes on the substituted ring. The synthesis of the 2'-amino-2,5-dimethylbenzophenone (B) necessary for the prepn. of A offered considerable difficulties. 2-*p*-Toluenesulfonylaminophenyl *p*-xylol ketone, crystals from  $\text{EtOH-Et}_2\text{O}$  (1:2), m.  $107-8^\circ$ , was obtained by heating 70 g. 2-*p*- $\text{MeC}_6\text{H}_4\text{SO}_2\text{NHC}_6\text{H}_4\text{CO}_2\text{H}$  in 350 cc. com. *p*-xylene with 70 g.  $\text{PCl}_5$  1 hr. at  $120-5^\circ$ , filtering hot as soon as the evolution of  $\text{HCl}$  ceases, cooling quickly (whereupon the chloride seps. in fine needles), adding 90 g.  $\text{AlCl}_3$  in several portions in the course of 1 hr., with shaking and cooling, heating within 0.5 hr. to  $70^\circ$ , letting stand 2 hrs. at  $70-5^\circ$ , decomp. with ice, acidifying with  $\text{HCl}$  and removing the excess of xylene with steam; attempts to sapon. the compd. with concd.  $\text{H}_2\text{SO}_4$ , 80%  $\text{H}_2\text{SO}_4$ , concd.  $\text{H}_2\text{SO}_4$ -glacial  $\text{AcOH}$ , concd.  $\text{HCl}$  or by alkali fusion were all unsatisfactory, only very slight sapon. occurring even after long continued action and at high temps.; the relatively best results were obtained with 80%  $\text{H}_2\text{SO}_4$  and with  $\text{H}_2\text{SO}_4$ - $\text{AcOH}$  on heating. Next 2-*p*-xylol-*o*-benzamide, needles from aq. alc., m.  $149-50^\circ$ , prepd. from the acid through the chloride, was treated by the Graebe-Ullman method and its various modifications for the conversion of amides into amines, but there were always obtained very impure resinous masses from which  $\text{HCl}$  extd. only small amts. of oily  $\text{NH}_2$  ketone. Attempts to prep. *o*- $\text{O}_2\text{NC}_6\text{H}_4\text{COC}_6\text{H}_4\text{Me}$  from  $\text{O}_2\text{N}_2\text{CH}_2\text{COC}_6\text{H}_4$ , *p*-xylene and  $\text{AlCl}_3$  in  $\text{CS}_2$  gave only a resinified mass from which no homogeneous product could be isolated. Finally, 40 g. *o*- $\text{BrC}_6\text{H}_4\text{CO}_2\text{H}$  in 180 cc. com. *p*-xylene was heated 1 hr. at  $110^\circ$  with 35 g.  $\text{PCl}_5$ , brought to a boil, filtered hot, cooled, treated with 55 g.  $\text{AlCl}_3$ , heated 2-3 hrs. at  $70^\circ$ , decompd. with ice, acidified, freed from

xylene with steam, boiled out with acidified hot  $H_2O$ , digested several times with hot  $Na_2CO_3$  and allowed to stand in the ice chest; there was thus obtained 35 g. 2'-bromo-2,5-dimethylbenzophenone, leaflets from dil. alc., m.  $46^\circ$ , b.  $270-2^\circ$ , 23 g. of which, heated 7-8 hrs. at  $180-90^\circ$  with 0.5 g.  $CuSO_4$  in 90 cc. abs. alc. satd. cold with  $NH_3$ , gives 18 g. B as a light yellow oil which decomps. completely on distn. under 15 mm.; by frequent rubbing on clay with alc. and ether of the product reprecipitated from HCl, a small amt. was obtained as a pure light yellow solid, m. turbid  $76-8^\circ$ , clear  $88-90^\circ$ . The crude B (30 g.) in 240 cc. concd.  $H_2SO_4$  at  $0^\circ$  was treated with 300 g. of a satd. soln. of  $O_2NSO_3H$  in concd.  $H_2SO_4$ , allowed to stand a short time at room temp., poured in a thin stream upon ice (keeping the temp. below  $10^\circ$ ), treated as soon as the pptn. is complete with 15 g. dry Cu powder, heated several hrs. on the  $H_2O$  bath and the A extd. with  $Et_2O$ . The  $Et_2O$  ext. was intensely fluorescent, red-brown in transmitted, green in incident light; it was freed of the few g. of  $o-HOC_6H_4COC_6H_4Me_2$  by shaking several times with dil. NaOH, washed with  $H_2O$ , dried and evapd. and the residue fractionated, yielding chiefly a light yellow oil,  $b_{10} 255-8^\circ$  (15 g.) and a darker oil,  $b_{10} 258-62^\circ$  (7 g.); on standing both fractions (especially the latter) deposited long yellow needles sep'd. from the oil by pressing on clay; in this way, from 22 g. of the 2 fractions was obtained 5 g. A, m.  $108^\circ$ ; the yellow oil was identified as 2,5- $Me_2C_6H_3COPh$ . When 1.5 g. A is added in the course of 20 min. to 9 g. KOH and a few drops of  $H_2O$  at  $235^\circ$  and is heated a few min. longer at  $235-40^\circ$ , dissolved in 200 cc.  $H_2O$ , filtered and pptd. cold with HCl there is obtained 1.1 g. of 2,5-dimethyl-6-phenylbenzoic acid, needles from dil. alc., m.  $145^\circ$ , is not esterified by  $MeOH-HCl$ , regenerates A when allowed to stand 10-5 min. in excess of concd.  $H_2SO_4$ .

CHAS. A. ROULLER

Halogenalkylated aromatic amines. V. Preparation of aryl vinyl ethers. JULIUS v. BRAUN AND GEORG KIRSCHBAUM. Landwirtsch. Hochschule, Berlin. Ber. 53B, 1399-1408 (1920); cf. C. A. 14, 1542.—The hope that vinyl ethers could be prep'd. by starting [with  $\beta$ -halogenethylated aromatic amines (e. g.,  $BrCH_2CH_2NMePh$  (A)] replacing the halogen by RO, converting the resulting basic ether with MeI or  $Me_2SO_4$  into a quaternary salt and decomg. the latter according to the scheme  $ROCH_2CH_2NMe_2PhI + KOH \rightarrow ROCH:CH_2 + PhNMe_2$  was not fulfilled in the cases where R is an aliphatic residue; the quaternary salts react with extraordinary slowness and only to a small extent in this way; to a large extent they undergo deep-seated decompn. and at the same time regenerate the original basic ether,  $ROCH_2CH_2NMePh$ . When R is an aromatic radical, however, the desired reaction proceeds smoothly, also with thiophenols and dihydroxybenzenes. The introduction of the vinyl group into phenols has a marked and very irregular effect on their odors. *N*- $\beta$ -Naphthoxyethylmethylaniiline (B), obtained in 80% yield from  $\beta$ -naphthol in an equal wt. of  $Me_2CO$  boiled 8 hrs. with 1 mol. each of A and  $K_2CO_3$ , crystals from alc., m.  $75^\circ$ , b.  $200-5^\circ$  in the vacuum of a Hg pump, is only weakly basic, sol. only in a large excess of dil. acid; hydrochloride, needles from  $EtOH-Et_2O$ , m.  $150^\circ$ ; chloroplatinate, yellow flocks, decomps.  $128-91^\circ$ . Methiodide, obtained quant. by digesting B 2 hrs. on the  $H_2O$  bath with MeI, crystals from  $H_2O$ , m.  $137^\circ$ ; treated in boiling  $H_2O$  with enough 40% NaOH to make a 15% NaOH soln. and distd. with steam it decomps. both into  $PhNMe_2 + CH_3:CHOC_6H_5$  and B + MeOH. The  $PhNMe_2$  in the distillate is dissolved by adding dil. HCl; the  $\beta$ -naphthol vinyl ether (45-50%) remaining as a cryst. mass, m.  $63-4^\circ$ ,  $b_{10} 137^\circ$ , has an intense mandarin-peel odor, does not undergo the Claisen rearrangement into a vinyl-naphthol, does not smoothly add Br (simultaneous evolution of HBr and substitution taking place) and is hydrogenated by the Paal method with extraordinary slowness and incompleteness; it is more resistant to hydrolysis than aliphatic vinyl compds., the odor of AcH becoming perceptible only after long boiling with alkalis or dil. acids. *N*- $\beta$ -Caracryloxyethylmethylaniiline, rather viscous oil,  $b_{10} 220-1^\circ$ ; picrate hydrochloride, oily; methiodide, oily; methochloride chloroplatinate, voluminous ppt., m.  $90^\circ$ . Caracryl

*vinyl ether*, b. 217–8°, has an unpleasant odor hardly reminiscent of carvacryl Me and Et ethers. *N-β-Tetrahydro-β-naphthylmethylaniline*, b<sub>11</sub> 257–8°, m. 52–3°; *hydrochloride*, m. 163°; *picrate*, m. 122°; *methiodide*, cryst. powder from EtOH-Et<sub>2</sub>O, m. 123°. *Tetrahydro-β-naphthyl vinyl ether*, b<sub>11</sub> 121–2°, has a not unpleasant but faint odor, while the *allyl ether*, b<sub>11</sub> 158–61°, has a stronger and more pleasant odor. Equimol. proportions of *o*-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> and A in Me<sub>2</sub>CO with K<sub>2</sub>CO<sub>3</sub> or in alc. with NaOEt give both the mono- and di-ethers; when working in alc., on cooling after disappearance of the alk. reaction, most of the product seps. as a cryst. magma; H<sub>2</sub>O and NaOH are added and the mixt. is shaken and filtered; acidification and addition of Na<sub>2</sub>CO<sub>3</sub> to the filtrate ppts. *N-β-o-hydroxyphenoxymethylaniline* (85%), crystals from EtOH-H<sub>2</sub>O, m. 71°; *hydrochloride*, darkens 140°, m. 195°; *picrate*, orange leaflets from alc., m. 146°; *acetate*, oily; *methiodide*, leaflets from H<sub>2</sub>O, m. 135°. The portion of the original product insol. in NaOH is *pyrocatechyl bis-β-methylphenylaminoethyl ether* (yield, 20% under the above conditions but 80% when 2 mols. A are used), crystals from EtOH-H<sub>2</sub>O, m. 90–1°; *hydrochloride*; *picrate*, fine yellow powder, m. 103°; *methiodide*, crystals from H<sub>2</sub>O, m. 96°; towards HCHO, BzH, BrCN, etc., the base behaves just like PhNMe<sub>2</sub>; the dyes derived from it show no striking differences in shade from the PhNMe<sub>2</sub> derivs. *Pyrocatechyl monoovinyl ether*, obtained in 75% yield from the above methiodide, boiled a few min. with alkali, b. 213°, has a faint odor. *Divinyl ether*, prepd. like the β-C<sub>10</sub>H<sub>7</sub>OCH:CH<sub>2</sub>, b. 200–9°, has a sharp, not pleasant odor; yield about 50%. *N-β-Phenylmercaptoethylmethylaniline*, b<sub>11</sub> 222–4°, has practically no odor; *hydrochloride*, oily; *picrate*, yellow needles from alc., m. 155°; *methiodide*, oily; *methochloride chloroplatinate*, scales from H<sub>2</sub>O, m. 155°. The methiodide with alkali gives 90% of *phenyl vinyl sulfide*, almost colorless liquid, b. 200–1°, has a pleasant rooty and at the same time radishy odor, becomes slightly turbid on standing and develops an unpleasant odor. *N-β-Amyloxyethylmethylaniline* (C), obtained by heating A with Na in AmOH for 45 hrs., b<sub>11</sub> 154–6°, has a very faint odor, forms oily salts and a non-crystallizable methiodide; *methochloride chloroplatinate*, darkens 160°, decomps. 166°. Alkali decomps. the methiodide very slowly, yielding, together with C, a very small amt. of a *N*-free acid-insol. oil of ether-like odor. *N-β-Thiocyanoethylmethylaniline*, obtained almost quant. by boiling A with aq. alc. KSCN, faintly yellow, practically odorless liquid, b<sub>11</sub> 186° without rearrangement into the mustard oil; *methiodide*, crystals from H<sub>2</sub>O, m. 137–8°, very quickly decompd. by alkalies, steam carrying over an oil which in the first moment smells distinctly of radishes, while an unbearable mercaptan odor soon manifests itself; an Et<sub>2</sub>O ext. of the distillate yields a very low boiling substance with mercaptan properties and, almost quant., PhNMe<sub>2</sub>. The residue from the steam distn. contains S and a dark amorphous S compd. Apparently, therefore, there is first formed a mixt. of NCSCH:CH<sub>2</sub> and PhNMe<sub>2</sub>, but the former at once decomps. C. A. R.

Preparation of tropic acid from formylphenylacetic ester. JULIUS V. BRAUN, Landwirtschaftl. Hochschule, Berlin. *Ber.* 53B, 1409(1920).—McKenzie and Wood, who obtained only 16–30% yields of tropic acid (A) (C. A. 13, 2857) by Müller's method (C. A. 12, 2556), evidently did not have the right conditions—possibly their Al-Hg was only slightly active. The synthesis has been carried out frequently in v. B.'s lab. with results like the following: From 18 g. HCOCHPhCO<sub>2</sub>Et (B) 8.2 g. pure A (53%) and 7.8 g. unchanged B; from 30 g. B 11.5 g. A (44%) and 16 g. recovered B.

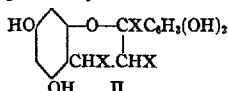
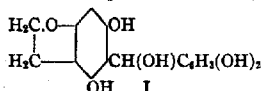
CHAS. A. ROVILLER

Keto-enol tautomerism. XII. Fractional distillation of acetoacetic ester. KURT H. MEYER AND VIKTOR SCHOELLER. *Akad. Wiss. München. Ber.* 53B, 1410–6 (1920); cf. C. A. 8, 1774.—If catalyzers are carefully excluded, mixts. of the keto and enol forms of AcCH<sub>2</sub>CO<sub>2</sub>Et can be sepd. without rearrangement by fractional distn. *in vacuo*; under certain other conditions the whole mixt. can be converted into 70–80% enol. *n*<sub>D</sub><sup>20</sup> was detd. for various mixts. whose enol % was measured by the Br titration

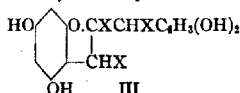
method, and a curve constructed by plotting the % of enol against  $n$ , and in this way the % of enol in the various fractions obtained in the distn. expts. below was detd. by measuring  $n$ . Using a Jena glass bulb of 30 cc. with a neck 40 cm. high and 5 cm. in diameter (the side arm being 30 cm. above the bulb) which had been steamed out, rinsed with alc. HCl and dried 3 hrs. at 100°, and distg. under 2 mm. ( $\text{CO}_2$ - and  $\text{NH}_3$ -free air being passed through the capillary), the four (equal) fractions of the distillate were found to contain 78, 76, 72 and 63% enol, resp., and the residue 5%. That the enolization does not take place in the vapor phase was shown by dropping mixts. of known enol content into a 1.2-m. Liebig condenser heated to 100°, so that the ester, under 2 mm., at once volatilized completely without fractionation. Starting with mixts. containing 23 and 7.4% enol, the vapors issuing from the app., when condensed in a freezing mixt., yielded liquids with the same % of enol as the original mixts. The enolization during distn. must, therefore, take place only in the liquid phase and not so much in the distg. bulb itself (in which an ester rich in ketone remains) as on the walls of the neck. On the other hand, in an app. of fused quartz, a mixt. containing 7.4% enol yielded 3 equal fractions with 22, 11 and 2.5% enol and a residue with 0%. Similarly, distn. of a mixt. of  $\text{BzCH}_2\text{CO}_2\text{Me}$  with 18% enol gave 3 fractions with 40, 21 and 20% enol and a residue with 8%.

CHAS. A. ROUVILLER

**Tannins.** V. Phloroglucinol tannins and catechols. Constitution of gambir catechol. KARL FREUDENBERG. Univ. Kiel. *Ber.* 53B, 1416-27(1920); cf. C. A. 14, 3413.—The behavior of the tannins in plants and a review of the catechol-like substances in nature lead to the conclusion that all the amorphous phloroglucinol tannins and their corresponding red condensation products are catechol-like compds. One way of testing the correctness of this hypothesis is to attempt to isolate the phloroglucinol-containing tannins as they are formed in the fresh leaves instead of in the brown-red, highly condensed and chemically indefinite forms in which they are present in the air-dried bark and wood; this is now being done with the native oak. Another way is the following: The flavone dyes, anthocyanidines and phenyl styryl ketones, show a very close parallelism with the catechols and pyrogallol tannins. Now, to the only representative of the catechols which has been well studied, viz., gambir catechol, Kostanecki has assigned a structure (I) differing markedly from that of the above



natural dyes, in which the two  $\text{C}_6\text{H}_4$  nuclei are joined by a bridge of three C atoms, and Perkin now also gives the preference to this structure (Perkin and Everest, "The Natural Organic Colouring Matters," London, 1918, pp. 438, 462). F. has now shown, however, by synthesis, that the product obtained by Kostanecki and Lampe by reduction of catechol tetra-Me ether with Na and alc. and subsequent methylation (C. A. 1, 1276) is 2,4,6,3',4'-pentamethoxy- $\alpha,\gamma$ -diphenylpropane (A); K.'s structure, therefore, is incorrect and gambir catechol is closely related, structurally, to the natural phloroglucinol dyes. F. assigns to it the structure II or III (two of the  $\text{X}'\text{s} = \text{H}$ , the other OH); he gives the preference to III, which explains the coumarane color reaction shown



by the tetra-Me ether. The veratric aldehyde (B) necessary for the synthesis of A was obtained in 21.5-22.5-g. yield by shaking and gently warming 20 g. 3,4-(HO)- $\text{C}_6\text{H}_3\text{CHO}$  with 20 cc. of 30% KOH and 18 cc.  $\text{Me}_2\text{SO}_4$ , adding 20 cc. KOH and 18 cc.

$\text{Me}_2\text{SO}_4$  three times more at about 5-min. intervals, finally 50 cc. more of KOH, shaking 10 min. longer, cooling, adding more KOH if the reaction is not alk. and at once extg. with a little  $\text{Et}_2\text{O}$ ; it dists. under 15 mm. at a bath temp. of  $200-20^\circ$  and solidifies completely, is somewhat sol. in cold  $\text{H}_2\text{O}$  and its satd. soln. gives with aq. tannin a milky ppt.; it is only very little volatile with steam.  $1,3,5\text{-C}_6\text{H}_3(\text{OH})_3$  (C) is obtained in 10.7 g. yield from 10 g.  $\text{C}_6\text{H}_3(\text{OH})_3$  (dried on the  $\text{H}_2\text{O}$  bath) in 100 cc. cold  $\text{MeOH}$  satd. with HCl, allowed to stand 48 hrs., concd. *in vacuo* to a sirup, heated with 40 cc.  $\text{Me}_2\text{SO}_4$ , treated in the course of 15 min. with 120 cc. of 7.5 *N* KOH at  $60-90^\circ$  and distd. with steam; the distillate is treated with concd. KOH and after some hrs. the crude C is filtered off, dried, freed from adhering oil on clay and crystd. from ligroin; 5 g. of this by Kostanecki and Tambor's method (*Ber.* 32, 2261 (1899)) yields 5.4 g. trimethylphloracetophenone, plates from alc., m.  $101-2^\circ$ , sol. in concd. HCl with yellow color, 5.3 g. of which with 5 g. B in 50 cc. alc. and 2.5 cc. of 10 *N* NaOH allowed to stand 24 hrs. at  $40^\circ$  gives 9 g.  $(\text{MeO})_3\text{C}_6\text{H}_2\text{COCH}:\text{CHC}_6\text{H}_4(\text{OMe})_2$ , crystals from alc., sol. in concd. HCl with violet-black color; this with Pt in AcOH quickly absorbs 6 atoms H, yielding A, very slender needle-shaped rectangles from alc., m.  $87-8^\circ$ . CHAS. A. ROUVILLE

**Diphenylaryltelluronium salts.** KARL LEDERER. *Prag. Ber.* 53B, 1430-45 (1920); cf. C. A. 14, 1988.—Triaryltelluronium salts are prepd. by quickly adding to 10 g.  $\text{Ph}_3\text{TeCl}_2$  in 200-50 cc. dry PhMe a freshly prepd. Grignard soln. (for the *o*- and *p*-tolyl compds. twice, for all the others three times the calcd. amt.), shaking thoroughly, quickly adding 15-20 cc. dil. HCl, dissolving the product in boiling  $\text{H}_2\text{O}$ , adding 10 g. KI in a little  $\text{H}_2\text{O}$ , filtering off the telluronium iodide after cooling, redissolving it in  $\text{H}_2\text{O}$  if it is colored, acidifying with a little AcOH, adding 2 g. KI and boiling for a long time whereby any basic iodide,  $\text{R}_3\text{Te}(\text{OH})\text{I}$ , decomps. into  $\text{R}_3\text{TeI}_2$  and  $\text{R}_3\text{Te}(\text{OAc})$ , the latter with the KI giving more of the former, which seps. as an intensely Bordeaux substance which can be easily filtered off. The triaryltelluronium iodide with alc.  $\text{HgI}_2$  gives the  $\text{HgI}_4$  double salt, with excess of AgCl in  $\text{H}_2\text{O}$  the chloride which in  $\text{H}_2\text{O}$  forms a  $\text{HgCl}_2$  double salt and with KBr the bromide; the picrates are obtained from the iodides or chlorides in  $\text{H}_2\text{O}$  with picric acid or Na picrate. *Diphenyl-p-tolyltelluronium iodide* (yield, almost 9 g.), cryst. powder of spherical aggregates from  $\text{H}_2\text{O}$ , microcolumns from  $\text{EtOH-Et}_2\text{O}$ , m.  $219-20^\circ$ ; *mercuric iodide compound*, microneedles from AcOH, softens  $220^\circ$ , m.  $222-3^\circ$ ; *bromide*, needles from  $\text{H}_2\text{O}$ , softens  $226^\circ$ , m.  $228-9^\circ$ ; *chloride*, partly cryst., partly a blistery mass; *mercuric chloride compound*, resinous mass from  $\text{H}_2\text{O}$ ; *picrate*, monoclinic prisms from alc., softens  $130^\circ$ , m.  $132-3^\circ$ . *Diphenyl-o-tolyltelluronium iodide* (yield, 5.1 g.), crystals from  $\text{EtOH-Et}_2\text{O}$ , softens about  $170^\circ$ , m.  $175-6^\circ$ ; *mercuric iodide compound*, amorphous powder, begins to sinter  $154^\circ$ , m. about  $184^\circ$ ; *chloride*, resinous; *mercuric chloride compound*, fine needles from AcOH, softens  $207-8^\circ$ , m.  $210-1^\circ$ ; *bromide*, cryst. powder from  $\text{H}_2\text{O}$  or  $\text{EtOH-Et}_2\text{O}$ , softens about  $199^\circ$ , m. about  $203^\circ$ ; *picrate*, triclinic microcolumns from alc., softens  $122^\circ$ , m.  $127-8^\circ$ . *Diphenyl-m-tolyltelluronium iodide*, needles from  $\text{H}_2\text{O}$ , softens  $190-1^\circ$ , m.  $190-1^\circ(?)$ , 4-sided microcolumns from alc., softens  $200^\circ$ , m.  $202^\circ$  (yield, 8.6 g.); *mercuric iodide compound*, crystals from alc., softens  $132^\circ$ , m.  $134-5^\circ$ ; *chloride* and *mercuric chloride compound*, resinous; *bromide*, needles from alc., softens  $199^\circ$ , m.  $202-3^\circ$ ; *mercuric bromide compound*, semi-solid; *picrate*, spherical aggregates of micropisms from alc., softens  $101^\circ$ , m.  $105-6^\circ$ . *Triphenyltelluronium iodide* (yield, 9.6 g.), softens  $245^\circ$ , m.  $247-8^\circ$ ; *mercuric iodide compound*, platelets from alc., turns brown  $146^\circ$ , begins to sinter about  $155^\circ$ , m. about  $178^\circ$ , becomes yellow in the light; *mercuric bromide compound*, long needles from alc., m.  $143-4^\circ$ ; *mercuric chloride compound*, 4-sided microcolumns from  $\text{H}_2\text{O}$ , softens  $130^\circ$ , m.  $136-7^\circ$ , silky needles from alc. or AcOH. *Diphenyl-o-xylyltelluronium iodide* (yield, 3.2 g.), microcubes with 2  $\text{EtOH}$  from  $\text{EtOH-Et}_2\text{O}$ , m.  $114-5^\circ$  (foaming), powder from  $\text{H}_2\text{O}$ , softens  $114^\circ$ , m.  $117-8^\circ$ . *Diphenyl-m-telluronium iodide* (yield, 5.8 g.), powder from alc., softens  $92^\circ$ , m. about  $103^\circ$ ; *mer-*



curic iodide compound, powder from alc., begins to sinter 195°, m. 201-2°. *Diphenyl-p-telluronium iodide* (yield, 5.3 g.), crystals from EtOH-Et<sub>2</sub>O, softens 211°, m. 213-4°; *mercuric iodide compound*, yellowish amorphous powder from alc., begins to soften 100°, m. about 110°; *chloride*, crystals from EtOH-Et<sub>2</sub>O, softens 207°, m. 210-1°; *mercuric chloride compound*, feldspar-like microcolumns from alc., softens 172°; m. 176°; *bromide*, columns from EtOH-Et<sub>2</sub>O, softens 218°, m. 220-1°; *mercuric bromide compound*, needles from AcOH or alc., softens 174°, m. 178-80°; *picrate*, yellow rhombic columns from alc., softens 168°, m. 170-1°. *Diphenyl-p-anisyltelluronium iodide* (yield, 10.2 g.), prisms with 0.5 mol. solvent from EtOH; *mercuric iodide compound*, amorphous powder from alc., becomes yellow on drying, softens 86°, m. 89-90°; the *chloride*, *bromide* and the corresponding *mercuric halide compounds* are oily or resinous; *picrate*, yellow monoclinic microcolumns from alc., softens 125°, m. 126-7°. *Diphenyl-o-anisyltelluronium iodide* (yield, almost 10 g.), crystals from EtOH-Et<sub>2</sub>O, softens 226°, m. 230-1°; *mercuric iodide*, leaflets of 4-sided elongated microplatelets from alc., softens 215°, m. 218-9°; the *chloride* and its *mercuric chloride compound* are resinous; *bromide*, crystals from EtOH-Et<sub>2</sub>O, softens 215°, m. 220-1°; *picrate*, lancet-shaped crystals from alc., softens 180-1°, m. 185-6°. *Diphenyl-m-anisyltelluronium iodide* (yield, 0.9 g.), cryst. meal from EtOH-Et<sub>2</sub>O, softens 85°, m. about 95°. *Diphenyl-p-phenyltelluronium iodide*, cryst. powder from EtOH-Et<sub>2</sub>O, softens 125°, m. about 131°; *mercuric iodide compound*, amorphous powder from alc., softens 74°, m. 76-7°. *Diphenyl-o-phenyltelluronium iodide* (yield, 8.1 g.), 4-sided columns from EtOH-Et<sub>2</sub>O, softens 225°, m. 247-8° (decompn.); *mercuric iodide compound*, scales from alc., turns lemon-yellow in the light, sinters 174°, m. 183-4°; the *chloride mercuric chloride compound* and *picrate* are waxy; *bromide*, cryst. powder from H<sub>2</sub>O, softens 174°, m. 178-9°. *Diphenyl-α-naphthyltelluronium iodide* (yield, 11.8 g.), cryst. meal from EtOH-Et<sub>2</sub>O, softens 130°, m. about 148°; *mercuric iodide compound*, powder, softens 115°, m. 126°; the *chloride* and *bromide* were not obtained pure. *Diphenylmesityltelluronium iodide* crystals from EtOH-Et<sub>2</sub>O, softens 152°, m. 153-4° (yield, 1.5 g.); *mercuric iodide compound*, powder from alc., begins to sinter 89°, m. 93-4°. CHAS. A. ROULLER.

*α-Naphtholphthalein* and its ethers. W. SCHULENBURG. Techn. Hochsch., München. Ber. 53B, 1445-57(1920).—*o*-(1,2-C<sub>10</sub>H<sub>6</sub>(OH)CO)C<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H (36 g.) in cold concd. H<sub>2</sub>SO<sub>4</sub> slowly stirred with 19 g. *α*-naphthol soon changes from red to deep blue and when poured upon ice, dissolved in Na<sub>2</sub>CO<sub>3</sub> to free it from small amts. of naphthol and naphthofluoran, pptd. with AcOH, dried at 100° *in vacuo* and crystd. from alc. and AcOEt 10-5 times yields 60-70% *o,o'*-*α*-naphtholphthalein (B) in colorless crystals, m. 253-4°, sol. in H<sub>2</sub>O, alc., AcOEt with yellow color, disappearing on addition of acids, in very dil. alkalis with green, in more concd. alkalis and in concd. H<sub>2</sub>SO<sub>4</sub> with deep blue color, thus agreeing in its properties with the product described by Csányi as the *p,p'*-compd. (C. A. 14, 1549). That the HO in the second naphthol residue is really in the *o*-position to the methane C atom is shown by the fact that B when heated a short time above its m. p. loses H<sub>2</sub>O and forms *α*-naphthofluoran. B is also obtained, but in small yield (8-10%), by the method hitherto used by other investigators, *viz.*, the condensation of phthalyl chloride with *α*-naphthol. No sign of the formation of a *p*-isomer was observed; to be sure, there was formed a substance sol. in alkalis with deep green color, as mentioned by Grabowsky (Ber. 4, 726(1871)), but this proved to be A, which, when pure, dissolves in alkalis with intense yellow color but when contaminated with traces of B gives green solns. 1,4-Naphthanisolephthaloylic acid (C), C<sub>10</sub>H<sub>6</sub>(OMe)COC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, however, is obtained in 73% yield when 26 g. *α*-C<sub>10</sub>H<sub>6</sub>OMe, 25 g. C<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>O and 180 g. CS<sub>2</sub> are slowly treated with 44 g. AlCl<sub>3</sub>, allowed to stand 1 day and warmed 1 hr. on the H<sub>2</sub>O bath; it seps. from alc. in stout prisms, m. 194°; *sodium salt*, rhombic tables from H<sub>2</sub>O; *calcium salt*, pearly prisms with 10 H<sub>2</sub>O. C is also obtained in 85% yield from 25 g. C<sub>10</sub>H<sub>6</sub>OMe, 32 g. phthalyl chloride

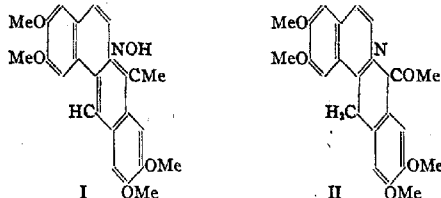
and 50 g.  $\text{CS}_2$  gradually treated with 4 g.  $\text{AlCl}_3$  and poured into ice- $\text{H}_2\text{O}$  after 45 min. That C is really a 1,4-compd. is indicated by the fact that all efforts to demethylate it failed; 5 g. heated in  $\text{AcOH}$  with 20 g.  $\text{HBr}$  (48%) 1.5 hrs. at  $100^\circ$  gave unchanged  $\text{C}$ ,  $\text{C}_{10}\text{H}_7\text{OMe}$  and phthalic acid; at higher temps. some naphthol was also formed. Again, there was no reaction when 12 g. C was heated with 8 times the calcd. amt. of  $\text{AlCl}_3$  4 hrs. in boiling  $\text{CS}_2$  or in  $\text{PhNO}_2$  at  $100^\circ$ . *p-Naphtholphthalalein dimethyl ether* (D) (see below) behaved in the same manner. From 16 g. C added to 220 g.  $\text{KOH}$  and 22 g.  $\text{H}_2\text{O}$  at  $220^\circ$  and heated 5 min. at  $250^\circ$  are obtained  $\alpha$ -naphthol and 82% of 1,4- $\text{C}_{10}\text{H}_6(\text{OH})\text{CO}_2\text{H}$ . Normal methyl ester of C, prepd. with  $\text{Me}_2\text{SO}_4$ , rhombic tables from alc., m.  $96^\circ$ ;  $\psi$ -ester, prepd. with  $\text{SOCl}_2$  and abs.  $\text{MeOH}$ , needles from  $\text{AcOEt}$ , m.  $120^\circ$ . A with twice the calcd. amt. of  $\text{Me}_2\text{SO}_4$  gives the methyl ester, double pyramids from alc., m.  $117-9^\circ$ , of the 1,2-isomer of C, needles from  $\text{C}_6\text{H}_6$ , m.  $179^\circ$ . The above results are not in harmony with the statements of Copisarow and Weizmann (*C. A.* 9, 2519) that the condensation of phthalyl chloride with  $\alpha\text{-C}_{10}\text{H}_7\text{OEt}$  yields first 1,2-ethoxynaphthol-*o*-benzoic acid (E) and then the di-Et ether of B. Accordingly, phthalyl chloride was condensed with  $\text{C}_{10}\text{H}_7\text{OEt}$  as in the prepn. of C; the 1,4-ethoxynaphthyl-*o*-benzoic acid (F) so obtained, pointed rectangular prisms from  $\text{PhMe}$ , m.  $166-7^\circ$  (C. and W. give  $155-6^\circ$  for their product), was as unreactive towards  $\text{AlCl}_3$  as C, behaved towards concd.  $\text{H}_2\text{SO}_4$  just like C (orange-red in the cold, red after 15 min. in the cold or 45 min. at  $80^\circ$ , darker after 15 min. at  $100^\circ$ ) and on fusion with alkali yielded 1,4- $\text{C}_{10}\text{H}_6(\text{OH})\text{CO}_2\text{H}$ . The product of further action, insol. in alkalis (probably the di-Et ether of the naphtholphthalalein), did not m.  $298^\circ$ , while C. and W. say their product m.  $159^\circ$ . The true B, dull yellow stout prisms from  $\text{C}_6\text{H}_6$ , m.  $145^\circ$ , was obtained like the 1,2-isomer of C with  $\text{Et}_2\text{SO}_4$  through its ethyl ester, prisms from alc., m.  $89^\circ$ . C is only feebly acidic; its Na salt is to a large extent decompd. by  $\text{CO}_2$  in  $\text{H}_2\text{O}$  and the free C begins to sep. from its soln. in 0.1 *N*  $\text{NaOH}$  on addition of 0.1 *N*  $\text{HCl}$  while the soln. is still alk. to phenolphthalein; this behavior can be explained on the assumption that the C tautomerizes into the hydroxyphthalide form; the 2 forms can be isolated as the esters (see above). All attempts to condense C to a naphthanthraquinone deriv. with  $\text{H}_2\text{SO}_4$  failed; under mild conditions only the vivid red lactone form resulted and with more vigorous treatment  $\text{SO}_3\text{H}$  acids were produced. Heated above its m. p., C decomp.; among the products were identified  $\text{C}_{10}\text{H}_7\text{OMe}$ , phthalic acid and  $\alpha$ -naphthofluoran and there was also isolated an alkali-insol. substance sepg. from  $\text{AcOEt}$  in faintly yellowish hexagonal prisms, m.  $201-2^\circ$ , turning red with concd.  $\text{H}_2\text{SO}_4$ . D, obtained in 24-g. yield from 20 g. C and 10 g.  $\text{C}_{10}\text{H}_7\text{OMe}$  slowly treated, with cooling and stirring, with concd.  $\text{H}_2\text{SO}_4$  and poured into ice  $\text{H}_2\text{O}$  after 2 hrs., fine needles from  $\text{AcOEt}$ , m.  $246-7^\circ$ , dissolves only with difficulty in alc.  $\text{KOH}$  even on boiling, dissolves with intense green color in concd.  $\text{H}_2\text{SO}_4$  or in  $\text{HCl}$  or  $\text{HBr}$  in  $\text{AcOH}$ ; it is also formed in 87% yield from phthalyl chloride and 2 parts  $\text{C}_{10}\text{H}_7\text{OMe}$  allowed to stand several days in  $\text{CS}_2$  with a trace of  $\text{AlCl}_3$ . The *o*-isomer, from B and 4 times the calcd. amt. of  $\text{Me}_2\text{SO}_4$  treated with anhydrous  $\text{Na}_2\text{CO}_3$  and warmed until a powdery mixt. results, then warmed with  $\text{Na}_2\text{CO}_3$  soln. until permanently alk., microneedles from dil. alc. or glacial  $\text{AcOH}$ , m.  $291^\circ$ , sol. in  $\text{H}_2\text{SO}_4$  with deep crimson color. CHAS. A. ROUILLER

Remarks on the work of Walter Fuchs and Benno Elsner: Tautomerism of phenols. II. Resorcinol. HANS TH. BUCHERER. Techn. Hochsch., Charlottenburg. *Ber.* 53B, 1457-9 (1920).—F. and E. conclude (*C. A.* 14, 3246) that the product formed from 1 mol.  $m\text{-C}_6\text{H}_4(\text{OH})_2$  and 3 mols.  $\text{NaHSO}_3$  is the bisulfite compd. of 3,5-diketohexamethylene-1-sulfonic acid, whose formation can be explained on the "assumption that it is produced from the tautomeric form,  $\text{CH}:\text{CH}.\text{CO}.\text{CH}_2.\text{CO}.\text{CH}_3$ , of  $m\text{-C}_6\text{H}_4(\text{OH})_2$ ."

B. believes this assumption is entirely erroneous, for two reasons: (1) The striking stability of the substance towards boiling dil. mineral acids; according to all his ex-

perience ketone-bisulfite compds. are decompd. with extraordinary ease by mineral acids while all  $\text{H}_2\text{SO}_4$  esters of mono- and bivalent phenols (and their  $\text{SO}_3\text{H}$  acids) are stable towards boiling dil. acids. (2) By the action of  $\text{NaHSO}_3$  on  $\text{C}_6\text{H}_5\text{N}$  is obtained a compd.  $\text{C}_6\text{H}_5\text{N} \cdot 3\text{NaHSO}_3 \cdot 2\text{H}_2\text{O}$  (C. A. 2, 2250), in whose formation the  $\text{C}_6\text{H}_5\text{N}$  nucleus is deeply modified, for with dil. alkali in the cold the larger part of the pyridine N is liberated as  $\text{NH}_3$ ; hence compds. of cyclic systems with 2 or 3 mols.  $\text{NaHSO}_3$  are easily produced without its being necessary to presuppose the intervention of C:O groups. Although his connection with industrial matters has prevented him for many years from devoting himself to purely scientific problems, B. has by no means given up the subject of the sulfite reactions and requests that the field be left to him. C. A. R.

**Acetopapaverine and coralyn (hexadehydrocoralydine).** WILHELM SCHNIEDER AND KURT SCHROETER. Univ. Jena. Ber. 53B, 1459-69 (1920).—When 8 g. concd.  $\text{H}_2\text{SO}_4$  is heated with 100 cc.  $\text{Ac}_2\text{O}$  several min. at  $85^\circ$  until a sample no longer gives a ppt. with  $\text{BaCl}_2$ , cooled, treated with 10 g. papaverine, again heated 15 min. at  $85^\circ$  and cooled there seps. 13.4 g. of the *sulfoacetate* (A),  $\text{C}_{22}\text{H}_{22}\text{O}_4\text{NSO}_3\text{CH}_2\text{CO}_2\text{H} \cdot \text{H}_2\text{O}$ , intensely yellow needles from  $\text{H}_2\text{O}$ -EtOH, m.  $277^\circ$ , forming intensely yellow-green fluorescent solns. in org. solvents, of an ammonium base, *hexadehydrocoralydine* (I), which



owing to its close structural relationship with Pictet's coralydine (C. A. 10, 1191) is designated as *coralyn*. In hot  $\text{H}_2\text{O}$  with concd.  $\text{HCl}$  A yields the *chloride*,  $\text{C}_{22}\text{H}_{22}\text{O}_4\text{NCl} \cdot 2.5 \text{H}_2\text{O}$ , undecompd. by  $\text{NaOH}$ . *Iodide*, from A in hot  $\text{H}_2\text{O}$  and  $\text{KI}$ , fine golden yellow needles with 1  $\text{H}_2\text{O}$ , m.  $278^\circ$  (decompn.), sol. in about 1600 parts  $\text{H}_2\text{O}$  at  $100^\circ$ , unchanged by  $\text{NaOH}$ . *Acid sulfate*, from hot aq. A and 50%  $\text{H}_2\text{SO}_4$ , light yellow voluminous ppt. with 2.5  $\text{H}_2\text{O}$ . *Nitrate*, from A in boiling  $\text{H}_2\text{O}$  and  $\text{HNO}_3$  (1:5), hair-like felted yellow needles, decomps. above  $270^\circ$ . When A in  $\text{H}_2\text{O}$  is treated with a few drops of dil.  $\text{NaOH}$  there is no change; on addition of more alkali the soln. darkens, finally to a black-brown color, and now probably contains the base I; a large excess of concd. alkali ppts. amorphous light yellow flocks, very probably of the pseudo base II (see above), for on diln. with  $\text{H}_2\text{O}$  they gradually redissolve with dark color. Attempts to obtain the pseudo base by means of  $\text{Et}_2\text{O}$  (Haars, Arch. Pharm. 243, 170 (1905)) failed on account of the insoly. of the ppt. in  $\text{Et}_2\text{O}$ . Attempts to obtain a  $\text{CHCl}_3$  or  $\text{Me}_2\text{CO}$  compd. of I likewise did not succeed. If a suspension of A in aq. alc. is treated with alc.  $\text{KOH}$ , A dissolves, at first with a dark color which gradually disappears. Addition of much  $\text{H}_2\text{O}$  to the almost colorless, not markedly fluorescent soln. (which probably contains the pseudo base) produces after a few sec. an intense yellow color and fluorescence, due to regeneration of the ammonium base. The pseudo base, *acetopapaverine* ( $\psi$ -*coralyn*) (II), obtained in 1.3-g. yield from 2 g. A in 50 cc.  $\text{H}_2\text{O}$  and 15 cc. of 5%  $\text{NaOH}$  quickly heated over a free flame to incipient boiling, seps. as an oil, solidifying on cooling to a yellowish gray cryst. mass, m.  $140-1^\circ$ , insol. in  $\text{H}_2\text{O}$  but gradually converted in contact with it into the sol. I which then imparts an alk. reaction to the soln.; in cold  $\text{MeOH}$  it dissolves in small amt. with light yellow color; diln. with  $\text{H}_2\text{O}$  does not reppt. it and at first the soln. is neutral to phenolphthalein, but after some min. becomes alk.; by titration with 0.1 N acid the velocity of rearrangement into I

can be followed easily. If, on the contrary, II is dissolved in hot MeOH or EtOH, the resulting dark brown solns. probably contain considerable I, as they at once give an alk. reaction on diln. with H<sub>2</sub>O. A stable soln. of II is easily obtained by heating with well-dried C<sub>6</sub>H<sub>6</sub>. Oxime, obtained quant. from 2 g. A in 15 cc. of 5% NaOH treated with excess of NH<sub>2</sub>OH.H<sub>2</sub>SO<sub>4</sub>, then with more NaOH until the dark color of the original soln. reappears, boiled and pptd. with CO<sub>2</sub>, crystals from 50% MeOH, m. 207°. Phenylhydrazone, from II heated with the least possible amt. of PhNHNH<sub>2</sub>, and treated still warm with alc. NaOAc, felted needles from MeOH, m. 208°. Methiodide, from 1.1 g. II heated with MeI in 10 cc. C<sub>6</sub>H<sub>6</sub> at 100° (with MeI in the presence of MeOH only the iodide of I is obtained), faintly yellowish needles from MeOH, m. 219–20°. Oxidation of A with alk. KMnO<sub>4</sub> (cf. Pictet and Malinowsky, C. A. 8, 101) yields only meta-hemipinic acid, identified as the ethylimide, m. 228°. CHAS. A. ROUILLER

**Synthetic preparation of humic acids.** WILHELM ELLER AND KATE KOCH. Univ. Jena. Ber. 53B, 1469–76 (1920).—It is shown that the deep black-brown products formed by the oxidation of phenols in alk. soln. are humic acids; apparently, however, only such phenols yield humic acids as can in some way pass through a quinoid stage as an intermediate product; thus, *m*-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> forms no humic acid. The products obtained from *o*- and *p*-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> and quinone have the compn. C<sub>8</sub>H<sub>4</sub>O<sub>4</sub>. As they do not cryst. and cannot be purified by repptn. their compn. can be confirmed only by the agreement of analyses on numerous different preps. The proof of their identity with the natural humic acids is likewise difficult, but the agreement in all physical properties and chem. reactions is so perfect that there remains no doubt as to such identity. The oxidation of the phenols to humic acids was effected more easily with K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> in alk. soln. than with atm. O. As E. and K. have now had in their possession homogeneous pure humic acids, they feel justified in making the following statements in regard to the chemistry of these substances: The compn. C<sub>8</sub>H<sub>4</sub>O<sub>4</sub> is materially simpler than has hitherto been assumed. While no method of detg. their mol. wt. has yet been found, it is, in view of their colloidal nature, probably high. It can be said with some certainty that there is not only one humic acid, but that different phenols yield different humin substances, especially since the intermediate product in the oxidation of bivalent phenols is certainly the corresponding quinone, for the quinol humic acid can be obtained equally well from quinone as from quinol, and if not enough alkali is used in the oxidation of quinol quinone can always be detected. The present work offers new proof that the N of natural humins is due to impurities; it sheds light on the formation of humins from sugar (Bottomley, C. A. 9, 3253), which may now be represented by the scheme C<sub>6</sub>H<sub>12</sub>O<sub>6</sub> + O = 4H<sub>2</sub>O + C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>, the first step possibly being a dehydration to quinone, C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>; and finally it shows that the acid reaction of humin substances is not due to adsorbed impurities; the humic acids are true acids whose acid reaction is due to unchanged phenolic HO groups. The humic acid from quinol is obtained in 60–70% yield by treating 5 g. quinol in excess of very dil. NaOH with 25 g. K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> in the course of 1 hr., cooling, slowly adding dil. HCl until the supernatant liquid has become light in color, filtering, washing with H<sub>2</sub>O until free from chlorides (at least 2, usually 5–6 days), and drying 5–6 hrs. at 80° and finally 7–8 days *in vacuo* over H<sub>2</sub>SO<sub>4</sub>; so obtained it is a brownish black, brittle, lustrous but amorphous powder, infusible, burning with a shower of sparks at higher temps., easily sol. in alkalies, easily but slowly in concd. H<sub>2</sub>SO<sub>4</sub>, also easily sol. when not fully dried in H<sub>2</sub>O and AcOH. It is also obtained, but in smaller yield, from quinol in dil. alkali exposed to the air in flat vessels for 6–7 days. The humic acids from quinone and *o*-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> are similarly obtained. PhOH in alkali treated with K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> as above gives products differing from the other synthetic humic acids only in their greater soly. in alc. Among the properties in which the natural and synthetic humic acids agree may be mentioned: (1) Compo. (2) Soly. in H<sub>2</sub>O and alc. and in traces in Et<sub>2</sub>O when freshly pptd. and in-

soly. when dry in these solvents; moreover, when pptd. by acids and washed, they gradually become sol. as the acid is replaced by the wash waters, and the last trace of the pptg. acid can be removed only with the greatest difficulty. (3) Salt formation; the alkali salts are easily, the others difficultly sol.; the  $\text{Fe}^{++}$  differs from the Ag salt in not dissolving on washing. (4) Chemical reactions: Cl gives a white product,  $\text{HNO}_3$  a deep red soln. yielding a brown amorphous ppt. when poured into  $\text{H}_2\text{O}$ , Br a white ppt. of  $\text{CBr}_4$  and also  $\text{CHBr}_3$ ;  $\text{Ac}_2\text{O}$  seems partially to acetylate the humic acids.

CHAS. A. ROUILLER

Preparation of  $\alpha$ -derivatives of hydroxylamine by means of potassium hydroxylaminedisulfonate.  $\alpha$ -Methylhydroxylamine and  $\alpha,\alpha$ -ethylenedihydroxylamine. WILHELM TRAUER, HEINRICH OHLENDORF AND HERBERT ZANDER. Univ. Berlin. *Ber.* 53B, 1477-92 (1920).—The basic salt  $\text{KON}(\text{SO}_3\text{K})_2$  obtained from  $\text{HON}(\text{SO}_3\text{K})_2$  and KOH (Raschig, *Ann.* 241, 183) reacts very easily with dialkyl sulfates and alkyl halides (it is not necessary to isolate the salt; the aq. soln. obtained by dissolving  $\text{HON}(\text{SO}_3\text{K})_2$  (A) in the calcd. amt. of KOH can be used equally well) to form compds. of the type  $\text{RON}(\text{SO}_3\text{K})_2$  which, by warming with acids, can be decompd. successively into the compds.  $\text{RONHSO}_3\text{K}$  and  $\text{RONH}_2$ . Thus, dipotassium  $\alpha$ -methylhydroxylamine- $\beta,\beta$ -disulfonate (B) is obtained in rectangular tables in 80% yield from A in 1 mol. of 6% KOH shaken about a day with 1 mol. MeI (until  $\text{BaCl}_2$  and  $\text{NH}_4\text{OH}$  no longer give a voluminous ppt. of  $\text{Ba}[\text{ON}(\text{SO}_3\text{K})_2]_2$ ), filtered, concd. on the  $\text{H}_2\text{O}$  bath to crystn., and recrystd. from dil.  $\text{NH}_4\text{OH}$ ; it is obtained much more quickly from A in about 1.5 mols. KOH with  $\text{Me}_2\text{SO}_4$ , the energetic reaction being moderated by cooling. When just brought to a boil in dil.  $\text{H}_2\text{SO}_4$  freed from  $\text{H}_2\text{SO}_4$  with  $\text{Ba}(\text{OH})_2$  and from excess of the latter with  $\text{CO}_2$  and concd. it yields potassium  $\alpha$ -methylhydroxylamine- $\beta$ -sulfonate, pearly tables, which, freed of most of the K with the calcd. amt. of  $\text{HClO}_4$  and concd., yields the free acid as a sirup slowly crystg. in vacuo over  $\text{H}_2\text{SO}_4$ ; barium salt, long prisms with  $1\frac{1}{2}\text{H}_2\text{O}$ . When B is boiled in dil.  $\text{H}_2\text{SO}_4$  until a sample, freed from all  $\text{H}_2\text{SO}_4$  with  $\text{BaCl}_2$ , no longer gives a further ppt. of  $\text{BaSO}_4$  with  $\text{NaNO}_2$  and  $\text{BaCl}_2$ , and is then evapd. to dryness, alc. exts. from the residue a mixt. of the acid and neutral sulfates of  $\alpha$ -methylhydroxylamine (C), which can be sepd. by repeated crystn. from alc. To obtain C rapidly, the soln. of  $\text{KON}(\text{SO}_3\text{K})_2$ , after methylation, is boiled directly with  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$ , made alk. and boiled and the C which dists. over is received in  $\text{HCl}$ ; yield of  $\text{HCl}$  salt, about 80%. The free C is a mobile liquid of amine-like odor, b.  $49-50^\circ$ , reduces cold  $\text{NH}_4\text{-AgNO}_3$  but not Fehling soln.; neutral sulfate, scales from dil. alc., m.  $144^\circ$ ; nitrate, deliquescent prisms, detonates violently at  $300^\circ$ ; picrate, m.  $175^\circ$ . The  $\text{HCl}$  salt with the calcd. amt. of alkali and 1 mol.  $\text{BzH}$  yields in a few hrs. *anti*- $\text{PhCH:NOME}$ , m.  $191^\circ$ ; with the calcd. amt. of alloxan in  $\text{H}_2\text{O}$  is obtained Me violurate, m.  $258-65^\circ$ ; the  $\text{HCl}$  salt with  $\text{KCNO}$  in hot  $\text{H}_2\text{O}$  gives methoxyurea, long prisms from alc., m.  $82-3^\circ$ ;  $\text{PhNCS}$  yields *N*-phenyl-*N'*-methoxythiourea, m.  $116^\circ$ ; 2 mols. C in 6 parts  $\text{Et}_2\text{O}$  slowly treated with 1 mol.  $\text{ClCO}_2\text{Et}$  gives  $\text{MeONHCO}_2\text{Et}$ , b.  $186-8^\circ$ . When 2 mols. C in cold  $\text{Et}_2\text{O}$  is treated with 1 mol.  $\text{CS}_2$  there soon seps. an oil which, when the supernatant  $\text{Et}_2\text{O}$  is removed, sometimes crysts. in the cold; at room temp. it decomp. spontaneously with strong evolution of heat, but it is stable in alc., giving on diln. with  $\text{H}_2\text{O}$  and addition of  $\text{AgNO}_3$  an intensely yellow curdy ppt. almost insol. in cold dil.  $\text{HNO}_3$ , having approx. the compn. of silver methoxydithiocarbamate,  $\text{MeONHCS}_2\text{Ag}$ , and decomp. about  $40^\circ$  with evolution of vapors of penetrating odor; the original product is probably the salt  $\text{MeONHCS}_2\text{H.NH}_2\text{OME}$ . Dipotassium  $\alpha$ -ethylhydroxylamine- $\beta,\beta$ -disulfonate, from  $\text{KON}(\text{SO}_3\text{K})_2$  and  $\text{Et}_2\text{SO}_4$ , needles. Potassium  $\alpha$ -ethylhydroxylamine- $\beta$ -sulfonate, needles; free acid, hygroscopic.  $\text{EtONH}_2\text{HCl}$ , m.  $126^\circ$ . Dipotassium  $\alpha$ -ethylenedihydroxylaminedisulfonate, 8-sided elongated tablets. Tetrapotassium  $\alpha,\alpha$ -ethylenedihydroxylamine- $\beta,\beta,\beta,\beta$ -tetrasulfonate, crystals with  $2\text{H}_2\text{O}$  from dil.  $\text{NH}_4\text{OH}$ , is obtained in about

70% yield from 100 g. A in 360 cc. of *N* KOH boiled 1–1.5 hrs. with 50 g.  $(\text{CH}_2\text{Br})_2$ , slowly gives with  $\text{BaCl}_2$  a ppt. containing both K and Ba. Dipotassium  $\alpha, \alpha$ -ethylene-dihydroxylamine- $\beta, \beta$ -disulfonate, crystals with 2  $\text{H}_2\text{O}$ .  $\alpha, \alpha$ -Ethylenedihydroxylamine sulfate, from the tetrasulfonate converted by short boiling with very dil.  $\text{H}_2\text{SO}_4$  into the disulfonate, treated with the amt. of  $\text{HClO}_4$  necessary to combine with all the K in soln., concd., filtered from the  $\text{KClO}_4$ , somewhat dild., boiled until completely hydrolyzed, again concd. and pptd. with alc., m.  $260^\circ$ , forms an aq. acid soln. which decomps.  $\text{Na}_2\text{CO}_3$ ; hydrochloride, m.  $233^\circ$ ; nitrate, m.  $124^\circ$ , detonates above its m. p.; free base, from the salts with excess of concd. alkali extd. with  $\text{Et}_2\text{O}$  or  $\text{CHCl}_3$ , b.  $105^\circ$ , b. about  $203^\circ$  but easily explodes with violence, reacts only faintly alk. in  $\text{H}_2\text{O}$ . The sulfate with  $\text{KNCO}$  gives the diurea,  $(\text{CH}_2\text{ONHCONH})_2$ , crystals from  $\text{H}_2\text{O}$ , m.  $190^\circ$ , and with  $\text{PhNCS}$  in  $\text{CHCl}_3$  the bisphenylthiourea, crystals from alc., m.  $119^\circ$ . Dipotassium  $\alpha$ -benzenesulfohydroxylamine- $\beta, \beta$ -disulfonate, from  $\text{KON}(\text{SO}_3\text{K})_2$  and  $\text{PhSO}_3\text{Cl}$ , needles from very dil. alkali.  $\text{BrCN}$  or  $\text{ICN}$  with 1 or 2 mols.  $\text{KON}(\text{SO}_3\text{K})_2$  (with 2 mols. the yield may reach 80%) gives tetrapotassium iminocarbonyldihydroxylaminetetrasulfonate,  $\text{HN}:\text{C}(\text{ON}(\text{SO}_3\text{K})_2)_2 \cdot 2\text{H}_2\text{O}$ , needles; the normal product,  $\text{NCON}(\text{SO}_3\text{K})_2$ , is undoubtedly first formed and reacts with a second mol. of  $\text{KON}(\text{SO}_3\text{K})_2$ . C. A. ROUILLER

Alkyliminodisulfonic acids. WILHELM TRAUBE AND MAX WOLFF. Univ. Berlin. Ber. 53B, 1493–501 (1920); cf. preceding abstr.— $\text{KN}(\text{SO}_3\text{K})_2$  or its aq. soln. behaves towards dialkyl sulfates and alkyl halides like  $\text{KON}(\text{SO}_3\text{K})_2$  but the reaction is often not so smooth, probably because in solns. of the salt there is an equil.  $\text{HN}(\text{SO}_3\text{K})_2 + \text{KOH} \rightleftharpoons \text{KN}(\text{SO}_3\text{K})_2 + \text{H}_2\text{O}$ , and consequently two reactions may occur simultaneously with the alkylating agent, viz., alkylation of  $\text{KN}(\text{SO}_3\text{K})_2$  and action of the free KOH on the alkyl halide or sulfate. Dipotassium methylaminesdisulfonate, from 25 g.  $\text{HN}(\text{SO}_3\text{K})_2$  in 110 cc. *N* KOH shaken 1 hr. with 12.5 g.  $\text{Me}_2\text{SO}_4$ , leaflets, also obtained with  $\text{MeI}$  on the  $\text{H}_2\text{O}$  bath, converted by boiling up with very dil.  $\text{H}_2\text{SO}_4$  into potassium methylaminesulfonate. The disulfonate (25 g.) methylated as above and boiled directly with 25%  $\text{HCl}$  gives 5.9 g.  $\text{MeNH}_2 \cdot \text{HCl}$ . Dipotassium ethylaminesdisulfonate, obtained with  $\text{Et}_2\text{SO}_4$  after 1–2 hrs. or with  $\text{EtI}$  after 24 hrs. on the  $\text{H}_2\text{O}$  bath and freed from reformed  $\text{HN}(\text{SO}_3\text{K})_2$  and unchanged  $\text{KN}(\text{SO}_3\text{K})_2$  by pptn. of the basic Ba salt with  $\text{Ba}(\text{OH})_2$ , long needles. Ethylation of 20 g.  $\text{HN}(\text{SO}_3\text{K})_2$  and subsequent hydrolysis with  $\text{HCl}$  gives 4.1 g.  $\text{EtNH}_2 \cdot \text{HCl}$ . Dipotassium propylaminesdisulfonate, obtained in 40% yield after more than 24 hrs. boiling with  $\text{PrI}$ , long needles from  $\text{H}_2\text{O}$ ;  $\text{PrNH}_2 \cdot \text{HCl}$  is obtained in 40% yield by direct treatment of the  $\text{HN}(\text{SO}_3\text{K})_2$  with  $\text{PrI}$  and subsequent boiling with  $\text{HCl}$ . Dipotassium epihydrinaminesdisulfonate, obtained in a few hrs. at  $100^\circ$ . Tetrapotassium ethylenediaminetetrasulfonate is obtained in 3.6 g. yield, together with 2.9 g.  $\text{HN}(\text{SO}_3\text{K})_2$ , from 10 g.  $\text{KN}(\text{SO}_3\text{K})_2$  in 25 cc.  $\text{H}_2\text{O}$ , gently boiled about 6 hrs. with 3 g.  $(\text{CH}_2\text{Br})_2$ ; it seps. from very dil. alkali in needles, sol. to the extent of 0.2372 g. in 100 cc.  $\text{H}_2\text{O}$  at room temp., gives in very dil.  $\text{NH}_4\text{OH}$  with  $\text{BaCl}_2$  the dipotassium barium salt. Dipotassium ethylenediamine-*N, N'*-disulfonate, rectangular tablets; free acid, leaflets whose aq. soln. with  $\text{BaCl}_2$  remains clear in the cold for a long time, but on boiling gradually deposits  $\text{BaSO}_4$ . The tetrasulfonate (15 g.) on hydrolysis with  $\text{H}_2\text{SO}_4$  gives 3.7 g.  $(\text{CH}_2\text{NH}_2)_2 \cdot \text{H}_2\text{SO}_4$ . CHAS. A. ROUILLER

Some new transformations of ethylenediamine. WILHELM TRAUBE AND ELISABETH PEISER. Univ. Berlin. Ber. 53B, 1501–8 (1920).—Through  $\text{H}_2\text{NCH}_2\text{CH}_2\text{NHSO}_3\text{H}$  (A) (C. A. 14, 60) and its NO deriv.,  $\text{H}_2\text{NCH}_2\text{CH}_2\text{N}(\text{NO})\text{SO}_3\text{H}$  (B), it is possible to replace one  $\text{NH}_2$  group in  $(\text{CH}_2\text{NH}_2)_2$  by OH, OR or halogens without in any way altering the other  $\text{NH}_2$  group. The  $\beta$ -aminoethylsulfonitrosamidic acid (B), column-like crystals from  $\text{H}_2\text{O}$ , is obtained by gradually treating 2 g.  $\text{H}_2\text{NCH}_2\text{CH}_2\text{NHSO}_3\text{H}$  and 1.1 g.  $\text{NaNO}_2$  in cold  $\text{H}_2\text{O}$  with 14–5 cc. *N*  $\text{H}_2\text{SO}_4$ ; it is strongly explosive when dry, on warm days may decomp. spontaneously with violent detonation when dried on clay, also exploded by shock or friction. Boiled with  $\text{H}_2\text{O}$ , it decomps. quant. into  $\text{H}_2\text{NCH}_2$ -

$\text{CH}_3\text{OH}$ ,  $\text{N}_2$  and  $\text{H}_2\text{SO}_4$ ; slowly added to concd.  $\text{HCl}$  and evapd. to dryness it yields  $(\text{H}_2\text{NCH}_2\text{CH}_2\text{Cl})_2 \cdot \text{H}_2\text{SO}_4$ ; with  $\text{HBr}$  the reaction is so violent as to be explosive if dry **B** is used and the  $\text{HBr}$  is not previously cooled strongly; the product is  $\text{H}_2\text{NCH}_2\text{CH}_2\text{Br}$ . Added in small portions to cold 40%  $\text{HF}$ , evapd. to dryness, made alk. and distd. with steam, **B** gives  $\beta$ -fluoroethylamine, which has a strong  $\text{NH}_3$ -like odor and is very volatile with steam; the nitrate, hydrobromide and hydrochloride cryst. well but are exceedingly sol. and very hygroscopic; picrolonate, yellow needles, m.  $239^\circ$ . With boiling alc. **B** gives  $\text{H}_2\text{NCH}_2\text{CH}_2\text{OEt}$ .  $\beta$ -Aminoethyl methyl ether, from **B** and boiling  $\text{MeOH}$ , mobile liquid of strongly basic odor,  $b_{76} 95^\circ$ ; hydrochloride, hygroscopic crystals; picrolonate, yellow needles, m.  $235^\circ$ ; the  $\text{HCl}$  salt, evapd. with 1 mol.  $\text{KCNO}$ , gives  $\beta$ -carbamidoethyl methyl ether, crystals from  $\text{EtOH-Et}_2\text{O}$ , sinters  $61^\circ$ , m.  $63^\circ$ , while with  $\text{PhNCO}$  is obtained  $\beta$ -phenylcarbamidoethyl methyl ether, m.  $94.5^\circ$ .  $\beta$ -Aminoethyl propyl ether, from **B** and  $\text{PrOH}$ ; picrolonate, yellow prisms from alc., m.  $188^\circ$ . With  $\text{PhOH}$  **B** does not give the expected  $\text{H}_2\text{NCH}_2\text{CH}_2\text{OPh}$  but  $\text{H}_2\text{NCH}_2\text{CH}_2\text{OSO}_3\text{H}$ . C. A. ROUILLER

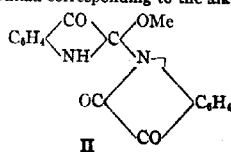
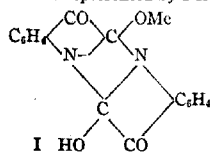
**Tautomerism of resorcinol.** J. HERZIG AND S. ZEISEL. Ber. 53B, 1518 (1920). —The interesting observation of Fuchs and Elsner (C. A. 14, 3246) is an extension of our knowledge of the tautomeric behavior of  $m\text{-C}_6\text{H}_4(\text{OH})_2$ , but by no means the first proof of it; cf. H. and Z., Monatsch. 10, 147 (1889); 11, 291 (1890). C. A. ROUILLER

**Tetranitromethane.** II. Tetranitromethane as a nitrating agent. I. ERICH SCHMIDT AND HEINRICH FISCHER. Univ. Berlin. Ber. 53B, 1529–37 (1920); cf. C. A. 13, 2362. —Tetranitromethane (**A**) is decompd. by aq. alkalis into  $\text{CO}_2 + \text{HNO}_3$ , on the one hand, and into  $\text{CH}(\text{NO}_2)_3 + \text{HNO}_3$  on the other; the latter decompn. becomes increasingly predominant with increasing concn. of the alkali and is the sole reaction when the alkali is sufficiently concd., also in the presence of weak alkali if it is carried out in alc. or  $\text{Me}_2\text{CO}$ ; in this sense  $p\text{-MeC}_6\text{H}_4\text{NMe}_2$  and  $\text{MeC}_6\text{H}_4\text{NEt}_2$  act as weak alkalis in alc., yielding in the cold  $4,2\text{-Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{NR}_2 \cdot \text{CH}(\text{NO}_2)_3$ ; i. e., the  $\text{HNO}_3$  set free acts as a nitrating agent. The  $\text{NO}_2$  group can sometimes be introduced into non-basic compds. by means of **A** by adding  $\text{C}_6\text{H}_5\text{N}$  to the reaction mixt. This method is the first which permits of introducing the  $\text{NO}_2$  group into the aromatic nucleus in an acid-free medium, and the H on C atoms with olefin double bonds can likewise be replaced by the  $\text{NO}_2$  group by this method. *m*-Nitro-*N*-dimethyl-*p*-toluidine-nitroform, yellow crystals from alc., m.  $91\text{--}2^\circ$  (decompn.), is obtained in 35-g. yield from 20 g.  $p\text{-MeC}_6\text{H}_4\text{NMe}_2$  in 20 cc. alc. and 9.2 g.  $\text{H}_3\text{BO}_3$  in 60 cc. alc. slowly treated in ice with 30.5 g. **A** in 20 cc. alc. and allowed to stand 2 hrs. in ice; it changes in 2–3 days to a brown oil; Sn and  $\text{HCl}$  reduce it to  $4,2\text{-Me}(\text{H}_2\text{N})\text{C}_6\text{H}_3\text{NMe}_2$ ; alkalis resolve it into its components; it can be synthesized from alc.  $\text{Me}(\text{O}_2\text{N})\text{C}_6\text{H}_4\text{NMe}_2$  and aq.  $\text{CH}(\text{NO}_2)_3$ . *m*-Nitro-*N*-diethyl-*p*-toluidine-nitroform (18.5 g. from 10 g.  $p\text{-MeC}_6\text{H}_4\text{NEt}_2$  and 3.8 g.  $\text{H}_3\text{BO}_3$  in 25 cc. alc. with 12 g. **A** in 10 cc. alc.), m.  $91^\circ$ , converted by alkalis into  $\text{CH}(\text{NO}_2)_3$  and *m*-nitro-*N*-diethyl-*p*-toluidine,  $b_{0.7} 101\text{--}2^\circ$  (yield, 82%). *m*-Nitro-*N*-dimethyl-*p*-anisidine-nitroform (6.3 g. from 3 g.  $\text{MeOC}_6\text{H}_4\text{NMe}_2$  and 1.2 g.  $\text{H}_3\text{BO}_3$  in 15 cc. alc. with 4.1 g. **A** in 5 cc. alc.), yellow crystals, decomp.  $108^\circ$ , converted by alkalis into  $\text{CH}(\text{NO}_2)_3$  and 90% of *m*-nitro-*N*-dimethyl-*p*-anisidine,  $b_{0.5} 108\text{--}10^\circ$ . When 5 g. *p*-cresol and 3.7 g.  $\text{C}_6\text{H}_5\text{N}$  in 10 cc. alc. are slowly treated cold with 9.5 g. **A** in 10 cc. alc., allowed to stand several hrs., filtered from the pyridine-nitroform,  $\text{C}_6\text{H}_5\text{N} \cdot \text{CH}(\text{NO}_2)_3$ , egg-yellow, decomp.  $91\text{--}2^\circ$ , and distd. with steam, there is obtained 4.3 g.  $4,2\text{-Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{OH}$ ,  $b_{0.5} 73\text{--}5^\circ$ , crystals from  $\text{AmOH}$ , m.  $33.5^\circ$ .  $\beta$ -Nitroisosafrrole, yellow needles from alc., m.  $98^\circ$ , is obtained in 72.5% yield from 8.1 g. isosafrrole and 4.8 g.  $\text{C}_6\text{H}_5\text{N}$  in 20 cc.  $\text{Me}_2\text{CO}$  treated with 9.8 g. **A** in 10 cc.  $\text{Me}_2\text{CO}$ , allowed to stand 2 hrs. in ice, dild. with  $\text{H}_2\text{O}$  and  $\text{Et}_2\text{O}$ , treated with 6.7 cc. of 33%  $\text{KOH}$  in 50 cc.  $\text{H}_2\text{O}$  in small portions, shaken until any oily  $\text{C}_6\text{H}_5\text{N} \cdot \text{CH}(\text{NO}_2)_3$  which may sep. has dissolved, sepd. from the aq. layer and evapd.  $\beta$ -Nitroanethole (64.3% from 7.4 g. anethole, 4.8 g.  $\text{C}_6\text{H}_5\text{N}$  and 9.8 g. **A** in  $\text{Me}_2\text{CO}$ ), crystals from  $\text{MeOH}$ , m.  $48^\circ$ . III. Conversion of

tertiary amines into secondary nitrosamines. I. *Ibid* 1537-44.—If the reaction between A and tertiary amines in the presence of  $C_2H_5N$  is carried out *hot* the  $HNO_3$  set free exerts no or very little nitrating action, but one of the alkyl groups on the N is split off as aldehyde and replaced by a NO group. This formation of nitrosamines with A is independent of the presence or of the position of other substituents on the  $C_6H_5$  nucleus. To 1 mol. of the tertiary amine and 1.2-1.5 mols.  $C_2H_5N$  in boiling alc. is slowly added 1.1 mols. of alc. A, the mixt. boiled to disappearance of the odor of A, shaken with about 100 cc.  $H_2O$  (for 10 g. amine), treated with 100 cc. satd. NaCl soln., extd. with  $Et_2O$ , the ext. treated with 100 cc. satd. NaCl and 2-3 g.  $K_2CO_3$ , distd. with steam, and the distillate treated with solid NaCl and extd. with  $Et_2O$ . In this way were obtained the following nitrosamines, with the % yields indicated:  $PhNMeNO$ ,  $b_{1.7}$  86-7°, 64% (from  $PhNMe_3$ ; as a by-product is formed  $p-O_2NC_6H_4NMeNO$ );  $PhNEtNO$ ,  $b_{1.4}$  96-7°, 40% (from  $PhNEt_3$ );  $o-MeC_6H_4NMeNO$ ,  $b_{1.4}$  89-90°, 90%;  $o-MeC_6H_4NEtNO$ ,  $b_{1.4}$  83-4°, 90%;  $o-ClC_6H_4NMeNO$ ,  $b_{1.2}$  89°, 92%;  $o-MeOC_6H_4NMeNO$ ,  $b_{1.1}$  112°, 44%; *N*-methyl-*m*-tolylnitrosamine, orange-yellow oil,  $b_{1.8}$  89-90°, 49%;  $m-ClC_6H_4NMeNO$ , m. 36-7°, 89%;  $p-MeC_6H_4NMeNO$ , m. 52°, 45%;  $p-ClC_6H_4NMeNO$ , m. 51°, 84%; *N*-ethyl-*p*-chlorophenylnitrosamine (B), felted needles from petr. ether, m. 60-1°, 95.5%;  $2,4-Me_2C_6H_3NMeNO$ ,  $b_{1.1}$  101°, 81%. The  $p-ClC_6H_4NEt$  used for the prepn. of B and described by Hofmann (*Ann.* 74, 144(1850)) as an oil non-volatile with steam is made by heating 40 g.  $p-ClC_6H_4NH_2 \cdot HBr$  and 22 g.  $EtOH$  10 hrs. at 150°, dissolving in a little  $H_2O$ , treating cold with a little HCl and concd.  $NaNO_2$ , freeing in the usual way from the mono-Et compd. by extg. with  $Et_2O$ , pptg. the di-Et base with KOH and taking up in  $Et_2O$ ; it  $b_{1.8}$  95-6° (yield, 29 g.) and seps. from alc. in needles, m. 39°.

CHAS. A. ROULLER

New isomerisms in the isatin series. IV. GUSTAV HELLER. Univ. Leipzig. *Ber.* 53B, 1545-51(1920); cf. C. A. 13, 2365.—As already shown (C. A. 11, 2778), isatol, the third isomer of isatin, has in boiling AcOH about 3 times the calcd. mol. wt. It has again been prepd. and crystd. from  $HCH(OMe)_2$ , from which it seps. in elongated prismatic crystals showing a mol. wt. of 398-9 in freezing veratrole, 405-68 in freezing PhOH; H., however, feels that this does not justify concluding that isatol is a polymeric product as it m. about 6° lower than isatin, but that it is merely a question of association. The conversion of isatol into isatin is not a simple rearrangement but apparently is effected through dimol. isatoid-like substances. If isatol is dissolved in alkali and acidified there sep. freely basic non-homogeneous flocks, sol. in concd. HCl and gradually sepg. again on addition of  $H_2O$ ; on attempting to recryst. them from  $HCH(OMe)_2$  a resin seps. along with crystals; they soften about 170° and m. about 175° (faint gas evolution). If the alk. isatol soln. is allowed to stand or is heated it is converted into the isatinone, apparently more smoothly on heating than in the cold; no formation of Friedländer's anhydro- $\alpha$ -isatinanthranilide is observed in sunlight. The structure assigned to isatinone (dimethylisatin III, C. A. 12, 2552) has been found to be incorrect; the substance is identical with Baeyer and Oekonomides' methylisatoid (*Ber.* 15, 2094). From the fact that it is converted in alk. soln. into anhydro- $\alpha$ -isatinanthranilide, H. believes it probably has the compn.  $C_{17}H_{17}O_4N_2$  (which agrees with the C values and one of the N values found by him; MeO found, 9.35), and that its structure can be represented by I or II, one formula corresponding to the alk. soln., the





other to the desmoptrope previously designated as "isatinol," a name which is now to be rejected and replaced by "isomethylisatoid." From 8.7 g. Ag chloroisatin heated 1-1.5 hrs. on the  $H_2O$  bath in 87 g.  $C_6H_6$  with 4.2 g.  $BzCl$  and filtered is gradually obtained 5-chloroisatol, crystals from  $AcOH$ , m.  $188^\circ$  (foaming), does not react with  $PhNHNH_2$ , nor with  $Me_2SO$ , on the  $H_2O$  bath, has no basic properties and is converted by hot alkalis into chloroisatinic acid. Chloroisatin lactim ether, crystals from  $C_6H_6$ , sinters about  $128^\circ$ , m. about  $132^\circ$ , yields with  $PhNHNH_2$  in alc. a dark red  $\alpha(2)$ -phenylhydrazosone, fine needles, from  $Me_2CO$ , m.  $250^\circ$ , which, when warmed with  $NaOH$  and Zn dust, yields a colorless filtrate becoming green-blue as the result of the formation of chloroindigo. The lactim ether can be converted into the chloromethylisatoid not only by illumination but also (although not homogeneously) by heating with  $AcOH$ . 5-Bromoisatol, obtained in 1.3 g. yield from 10 g. Ag 5-bromoisatin in 100 g.  $C_6H_6$ , heated 3 hrs. on the  $H_2O$  bath with 4.2 g.  $BzCl$ , crystals from  $Me_2CO$ , gives no indophenin reaction and dissolves in dil.  $NaOH$  with a light orange-yellow color, which gradually fades out. When 5 g. Ag bromoisatin is heated 2 hrs. at  $100^\circ$  with 4 g.  $MeI$  in 15 g.  $C_6H_6$ , and filtered hot there seps. after 24 hrs. 0.25 g. of a mixt. of needles and granules, the  $C_6H_6$  soln. of which with  $PhNHNH_2$  gives a red  $\alpha$ -phenylhydrazosone (A), indicating the presence of 5-bromoisatin lactim ether. The  $C_6H_6$  filtrate from the original mixt. when concd. on the  $H_2O$  bath and treated with several vols. of ligroin yields orange-red needles, m.  $172-3^\circ$ , giving the indophenin reaction, easily sol. in concd.  $HCl$  and in dil. alkali, sepg. unchanged on acidification, and thus characterized as *N-methyl-5-bromoisatin* (B); with  $PhNHNH_2$  it gives a yellow hydrazone. A seps. from alc. in dark red 4-cornered tablets with greenish metallic shimmer, decomp.  $241-2^\circ$ , gives a very faint violet indophenin reaction; when reduced with hot  $NaOH$  and Zn dust, the filtrate soon becomes bluish. B is obtained smoothly from 4 g. bromoisatin in 18 g. abs. alc. converted by means of 0.4 g. Na in 8 g. alc. into the Na salt, which is heated with 3-4 hrs.  $MeI$  2 hrs. at  $100^\circ$ .

CHAS. A. ROULLER

Thionaphthene in coal tar. R. WEISSGHRBER AND O. KRUBER. Ges. f. Teerverwertung m. b. H., Duisburg-Meiderich. Ber. 53B, 1551-65(1920).—W. and K. have found that thionaphthene (A) is present in all naphthalenes obtained from coal tar and have worked out relatively simple methods for isolating it from crude and from com. "pure"  $C_{10}H_8$ . Just as thiophene is difficult to sep. from  $C_4H_4$ , so A, in spite of its low m. p. and great soly., is found not in the liquid fractions of coal tar corresponding to its b. p. but in the solid  $C_{10}H_8$  fractions, even after the latter have been subjected to the com. methods of purification. If crude  $C_{10}H_8$  freed from all accompanying oil by pressing, is treated with an amt. of concd.  $H_2SO_4$  insufficient for complete sulfonation at  $90-100^\circ$  there is a considerable resinification of all those constituents of the crude product which, being unsatd. (like the indenenes and coumarones), are converted by  $H_2SO_4$  into polymerization products and resin acids. If the amt. of  $H_2SO_4$  is small (8-10% of the crude  $C_{10}H_8$ ), only a small part of the  $C_{10}H_8$  is converted into its  $SO_3H$  acids. It was this last waste product of  $C_{10}H_8$  purification which W. and K. used as their starting point. The concd. soln. obtained by washing the  $H_2SO_4$ -treated  $C_{10}H_8$  with small amts. of  $H_2O$  was decompd., after addition of  $H_2SO_4$ , with steam at about  $145^\circ$ , yielding a faintly yellowish cryst. mixt. of  $C_{10}H_8$  and A, together with small amts. of phenols and high boiling hydrocarbons, which, after treatment with  $NaOH$  and fractionation, m. about  $73^\circ$  and contains 2-3% S. If the process is repeated there is obtained an oily product with a S content of 5-6% which rises to 12-4% when the  $C_{10}H_8$  is frozen out. Continuation of this process probably would lead finally to pure A, but in view of the losses in each partial sulfonation, the process was stopped at this stage and attempts were made to isolate the A directly from the fractions rich in it. After many trials, it was found that 1 g. of the oil (with 14% S) in 20 cc.  $AcOH$  heated 0.5 hr. on the  $H_2O$  bath with 5 g. perhydrol and 5 cc.  $H_2O$ , then dild. with 100 cc.  $H_2O$

and allowed to stand several hrs. in the cold, gave 0.6 g. of A S-dioxide, m.  $142^{\circ}$  (Lanfray, C. A. 6, 1286). Pptr. as the  $\text{Hg}(\text{OAc})_2$  compd. is successful only if the temp. is moderated by using MeOH as solvent; 3 g. of the oil, b.  $218-24^{\circ}$ , with 14% S, in a freshly prepd. soln. of 4 g.  $\text{HgO}$  in 4 cc. AcOH and 30 cc.  $\text{H}_2\text{O}$  is boiled 1 hr. with 40 cc. MeOH, and the filtered  $\text{Hg}(\text{OAc})_2$  compd., after washing with alc. and  $\text{C}_6\text{H}_6$ , is decompd. under dil. HCl with steam, giving 0.8 g. pure A. By treating 80 kg. of com. "pure"  $\text{C}_{10}\text{H}_8$ , 0.5 hr. at  $100^{\circ}$  with 8 kg. of 95-6%  $\text{H}_2\text{SO}_4$  drawing off the acid, washing the  $\text{C}_{10}\text{H}_8$  twice with about 1%  $\text{H}_2\text{O}$ , removing any  $\text{C}_{10}\text{H}_8$  in the aq. soln. with PhMe, filtering off the  $\beta\text{-C}_{10}\text{H}_7\text{SO}_3\text{H}$  which crysts. out, decomp. with steam and treating the first 50 cc. of distillate with  $\text{Hg}(\text{OAc})_2$ , there were obtained about 2 g. pure A. Na reacts very energetically with A at high temps., rupturing the side ring and removing the S as  $\text{Na}_2\text{S}$ ; under certain, not easily maintained conditions, the reaction can be stopped at an intermediate stage and a Na compd. of A can be isolated which, on decompn. with  $\text{H}_2\text{O}$ , yields A; thus, 3 g. fused A slowly heated with 1 g. Na and kept at  $118-20^{\circ}$  until the reaction is complete, washed with  $\text{C}_6\text{H}_6$ , poured into ice  $\text{H}_2\text{O}$  and extd. with  $\text{Et}_2\text{O}$  yields 0.1-0.2 g. A; the aq. alk. soln. on acidification evolves much  $\text{H}_2\text{S}$  and deposits resinous amorphous products. When, however, A is heated with  $\text{NaNH}_2$ ,  $\text{NH}_3$  begins to be evolved at  $50-60^{\circ}$ ; at  $120^{\circ}$  the reaction is complete and sodium thionaphthene is obtained as an amorphous yellow-brown powder regenerating the A almost quant. with  $\text{H}_2\text{O}$ ; the alk. soln. does not give  $\text{H}_2\text{S}$  with acids. This reaction has been made the basis of a technical process for obtaining A from fractions enriched by the sulfonation process and even from pressed crude  $\text{C}_{10}\text{H}_8$ ; it has also been found that the  $\text{NaNH}_2$  can be replaced by free Na in a current of  $\text{NH}_3$  (Ger. pats., G. 48,631, 48,632). A treated in the usual way with Grignard reagents does not react either in the cold or on heating, but when 6.7 g. A in 20 cc.  $\text{PhNMe}_2$  is mixed with 3 g.  $\text{EtBr}$  and 1.2 g. Mg in 20 cc.  $\text{Et}_2\text{O}$ , whereupon heat is evolved and  $\text{C}_6\text{H}_6$  is liberated, then freed from the  $\text{Et}_2\text{O}$  by evapn., boiled gently for 1 hr., cooled, treated for several hrs. with dry  $\text{CO}_2$ , extd. with dil.  $\text{Na}_2\text{CO}_3$  and acidified there is obtained 2.1 g. thionaphthene-2-carboxylic acid (B), stout light brownish prisms from alc., fine needles from  $\text{H}_2\text{O}$ , m.  $236^{\circ}$ , mol. wt. in freezing AcOH 198. This behavior towards the Grignard reagent indicates that at least one H atom of the S ring of A can be replaced by metals. When 400 g. A in 500 cc. xylene is heated 6 hrs. with 300 g.  $\text{NaNH}_2$ , first at  $100^{\circ}$  and finally at  $145^{\circ}$ , then treated 12 hrs. at  $100-10^{\circ}$  with dry  $\text{CO}_2$ , poured into ice- $\text{H}_2\text{O}$ , sepd. from the xylene layer (which contains considerable unchanged or regenerated A) and acidified, and the ppt. (160 g.) is esterified in 6 parts MeOH by treatment for 3 hrs. on the  $\text{H}_2\text{O}$  bath with HCl, and the product is fractionated, there are obtained 60 g. of the methyl ester of B, light yellow oil of a faint but not pleasant odor,  $b_{14}$   $171^{\circ}$ , stout prisms from alc., m.  $72-3^{\circ}$  (ethyl ester, b.  $181-3^{\circ}$ , crystals from alc., m.  $36-7^{\circ}$ ), and 58 g. of dimethyl thionaphthene-2,3-dicarboxylate, long, stout prisms from alc., m.  $91^{\circ}$ ,  $b_{14}$   $213-5^{\circ}$ , converted by hot 40% KOH and a little alc. into the acid (C), m.  $250-1^{\circ}$  (Bezdrík, et al., C. A. 2, 1438), which, boiled 0.5 hr. with 3 parts  $\text{Ac}_2\text{O}$ , gives the anhydride, light yellow needles, m.  $171^{\circ}$ . B differs from the compd., m.  $114^{\circ}$ , described under the same name by Friedländer and Lenk (C. A. 6, 2923) and its constitution was accordingly confirmed as follows: 13 g. of the Me ester in 25 cc. alc. was heated 5 hrs. on the  $\text{H}_2\text{O}$  bath with an equal wt. of  $\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$ , giving 12.5 g. of the hydrazide, soft leaves from alc., m.  $184-5^{\circ}$ , 12 g. of which in 90 cc. cold AcOH with excess of  $\text{NaNO}_2$  gave the azide, long needles from alc., m.  $108^{\circ}$  (decompn.); this, rubbed to a paste with 2-3 times the calcd. amt. of abs. alc. and fused, yields 2-aminothionaphtheneurethan, yellowish sword-shaped crystals from alc., m.  $161^{\circ}$  (yield, 85%), which, when boiled a short time with AcOH and concd. HCl or heated 2 hrs. with concd.  $\text{NH}_4\text{OH}$  at  $180^{\circ}$ , smoothly gives 2-hydroxythionaphthene, m.  $34-5^{\circ}$  (Marschalk, C. A. 8, 1089). The chloride of B,  $b_{14}$   $173-5^{\circ}$ , leaflets from ligroin, m.  $88-9^{\circ}$ . Amide, fine needles from  $\text{H}_2\text{O}$ , sword-

shaped leaflets from alc., m. 177°. The 2- and 3-monoamides of C are easily formed by adding the powdered anhydride to 8-10 parts of MeOH satd. at 0° with  $\text{NH}_3$ ; after distg. off the alc. the resulting sirup is taken up in 10-2 parts of glacial AcOH and on cooling the 2-amide seps. in short needles, m. 221° (foaming) (yield, 50-70%); the concd. mother liquors, treated with a little  $\text{H}_2\text{O}$  and rubbed, give the 3-amide, short needles from alc., and AcOH, m. 193° (foaming) (yield, 30-50%); 2 g. of the 2-amide in the calcd. amt. of cold dil. NaOH heated 10 min. at 80-90° with 3.6 g. NaOH, 30 cc.  $\text{H}_2\text{O}$  and 1.7 g. Br, cooled, neutralized with  $\text{H}_2\text{SO}_4$ , acidified with AcOH, filtered and decompd. with  $\text{H}_2\text{SO}_4$  and steam gives 55% of 2-hydroxythionaphthene, while the 3-amide yields 50% of the 3-HO compd. *Thionaphthene-2,3-dicarboximide*, obtained in 60-70% yield from the above monoamides heated to 200-20° until the elimination of  $\text{H}_2\text{O}$  is complete and quickly distd. *in vacuo*, golden yellow leaflets from AcOH or alc., m. 236-7°, also obtained by heating the anhydride in  $\text{NH}_3$  or by fusing it with the calcd. amt. of urea; the Hofmann degradation yields exclusively (40%) 3-hydroxythionaphthene.

CHAS. A. ROUILLER

**Biphenylene sulfide in coal tar.** O. KRUBER. Ges. f. Teerverwertung m. b. H., Duisburg-Meiderich. Ber. 53B, 1566-7(1920); cf. preceding abstr.—From 10 g. crude phenanthrene, containing 1% S, in 150 cc. AcOH heated 40 min. on the  $\text{H}_2\text{O}$  bath with 30 g. perhydrol, dild. and cooled somewhat is obtained a brownish solid product, most of which easily dissolves in alc., leaving a sandy residue which also dissolves on long heating with much alc. and on cooling seps. in the long needles, m. 229-30°, of biphenylene sulfone.

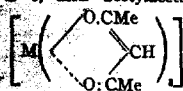
CHAS. A. ROUILLER

**Oxidation of hydrocarbons with oxygen. Oxidation of paraffin.** C. KILBER. Kraemer & Flammer, Heilbronn. Ber. 53B, 1567-77(1920); cf. C. A. 14, 2343.—The product obtained by the oxidation of paraffin consists of the distillate (chiefly fatty acids of low mol. wt., with ketones, alcs. and various organic compds., which were reported on in the last paper) and the residue, a yellowish mass with an odor reminiscent of the coconut fatty acids and an I no. of 1-2, sapon. no. of 250-300 and an acid no. of 200. This residue, which amts. to 90-100% of the original material, is the subject of the present paper. It was washed several times with warm  $\text{H}_2\text{O}$  to remove  $\text{H}_2\text{O}$ -sol. substances, sapond. with alkali, sepd. from unsaponifiable material, acidified with mineral acids, extd. with petr. ether and the acids in the ext. were converted into the Et esters and fractionated under 8-10 mm. The individual fractions were saponid. and as the acids could not be satisfactorily sepd. by crystn., recourse was had, for further sepn., to the differing soly. of their K salts in  $\text{Me}_2\text{CO}$ . This, too, however, did not effect a sepn. into the individual acids and Heintz's method (pptn. of the higher acids in alc. with  $\text{Mg}(\text{OAc})_2$ ) was applied to some of the fractions. Hehner and Mitchell's method for the detection of octadecanic (stearic) acid and that of Heiduschka and Burger for the sepn. of hexadecanic (palmitic) acid were also used. In the ester fractions  $b_1$  up to 120°,  $b_{8-10}$  120-200°,  $b_{9-10}$  200-30°, and  $b_{8-10}$  230-50° have thus far been identified the following straight-chain acids; capric, myristic, palmitic, heptadecanic, stearic and arachidic; and in the distillate:  $\text{HCO}_2\text{H}$ , AcOH,  $\text{EtCO}_2\text{H}$ ,  $\text{PrCO}_2\text{H}$ ,  $\text{BuCO}_2\text{H}$ ,  $\text{AmCO}_2\text{H}$ ,  $\text{C}_7\text{H}_{13}\text{CO}_2\text{H}$ ,  $\text{C}_8\text{H}_{17}\text{CO}_2\text{H}$  and  $\text{C}_9\text{H}_{19}\text{CO}_2\text{H}$ . In the ester fraction  $b_{8-10}$  120-200° was found an acid  $\text{C}_{16}\text{H}_{32}\text{O}_2$  isomeric with palmitic acid and possibly identical with Bergmann's "isopalmitic acid" (Z. angew. Chem. 1918 69). If paraffin is heated in O at 150-60° some time elapses before the "reaction" sets in with evolution of heat, whereas if the paraffin is previously heated for some time at 150-60° without O, the reaction at once sets in when O is introduced, showing that the action of the O is of a secondary nature and sets in only after the hydrocarbon has begun to decomp.

CHAS. A. ROUILLER

**Addition compounds in the acetylacetonates of the rare earths.** G. JANTSCH and E. MEYER. Techn. Hochschule Zürich und Karlsruhe. Ber. 53B, 1577-87(1920);

G. Bütz, *Ann.* 331, 334 (1904); B. and Clinch, *Z. anal. Chem.* 40, 218 (1904).—Since the data available indicate that the max. coordination number of the rare earth elements is 6, their acetylacetonates are doubtless coordinately satd. inner complex salts



$PhNH_2$ , and  $MeCN$  is conditioned by the fact that the acetylacetonate complex, which, as regards the rare earth element, is coordinately satd., still possesses as such free affinity, which is satd. by the additions in question, the addition products, therefore, being represented by the formula  $[MR_3] \dots R'$ , where  $R = -O.CMe:CH.CMe:O \dots$ .  $LaR_3$ , obtained in 6-g. yield from 3 g.  $La_2O_3$  by B.'s method, m.  $151^\circ$  after repeated crystn. (B. gives  $183^\circ$ ); 3 g. in 20 cc. cold abs. alc., treated about 10 min. with  $NH_3$ , and allowed to stand 1 hr. gives 2.5 g. of ammonia-lanthanum acetylacetonate,  $LaR_3 \cdot NH_3$ , fine needles, m.  $147.5^\circ$ , loses its  $NH_3$  completely on short boiling in alc. Pyridine compound, from 3 g.  $LaR_3$  heated over a free flame with 12 cc.  $C_5H_5N$  until it just dissolves and quickly filtered through a hot-water funnel, fine needles, m.  $142^\circ$ ; yield, almost quant. Aniline compound (3 g. from 3 g.  $LaR_3$ ), fine silky needles, m.  $192^\circ$ . Lanthanum dibenzoylmethane,  $La(C_6H_5CH_2O)_3$ , obtained in 5.5 g. yield from the  $La(OH)_3$  from 5 g. of the nitrate boiled in 40–50 cc. with 8 g.  $CH_3Bz_2$  in 100 cc. alc. until dissolved, dild. with  $H_2O$  until just turbid, heated on the  $H_2O$  bath until the turbidity again disappears, decanted from an oil which may have formed and allowed to cool slowly, seps. in light yellow needles, m.  $141-3^\circ$ ; 1 g. in cold alc. with  $NH_3$  gives 1 g. of the ammonia compound, fine faintly yellow needles, m.  $99^\circ$ , loses its  $NH_3$  on short standing in the air. Gadolinium acetylacetonate,  $CdR_3 \cdot 2H_2O$ , obtained in 4.5 g. yield from 5 g. of the nitrate in 10 cc.  $H_2O$  and 6 g.  $CH_3Ac_2$  in 5 cc. concd.  $NH_4OH$  and 30 cc.  $H_2O$ , seps. from abs. alc. in needles with 1  $H_2O$ , m.  $142^\circ$ ; 3.5 g. cautiously warmed with 15 cc.  $C_5H_5N$  until it dissolves (about  $60^\circ$ ) yields 2.6 g. of the pyridine compound, fine needles, m.  $139.5^\circ$ . Yttrium acetylacetonate,  $YR_3$  (9.5 g. from 3 g. of the oxide and 8 g.  $CH_3Ac_2$ ), needles from alc., m.  $131^\circ$ ; 5 g. in 30 cc. alc. with  $NH_3$  gives 4.6 g. of the ammonia compound, fine needles, m.  $129^\circ$ , loses its  $NH_3$  on long boiling in  $C_6H_6$ , converted by boiling with  $C_5H_5N$  into the pyridine compound (also obtained in 2-g. yield from 3 g.  $YR_3$  quickly heated with 10–2 cc.  $C_5H_5N$  until it dissolves), fine needles, m.  $121^\circ$ , which are converted by dry  $NH_3$  into the  $NH_3$  compd. Aniline compound (4.6 g. from 5 g.  $YR_3$  dissolved in 15 cc. hot  $PhNH_2$ ), needles, m.  $109^\circ$ . Acetonitrile compound (1.1 g. from 2 g.  $YR_3$  heated with 18 cc.  $MeCN$  until it dissolves), fine scales with a fatty luster, m.  $138^\circ$ , completely loses its  $MeCN$  on short standing in the air, more quickly in vacuo. Yttrium dibenzoylmethane, obtained in 4.8-g. yield by pptg. 5 g. of the nitrate in alc. with alc.  $NH_3$ , washing the hydroxide free of  $NH_3$  with alc., suspending in 40–50 cc. alc. and boiling with 3 mols.  $CH_3Bz_2$  in 70 cc. alc. until it dissolves, long yellowish needles, m.  $240^\circ$ ; no addition products with  $NH_3$ ,  $C_5H_5N$ ,  $PhNH_2$ , or  $MeCN$  could be obtained.

C. A. ROUILLE

Mustard oil preparation from non-aromatic primary bases. JULIUS V. BRAUN, Landwirthsch. Hochschule, Berlin. *Ber.* 53B, 1588 (1920).—In connection with Skita and Rolles' communication on cyclohexyl mustard oil (*C. A.* 14, 3667), v. B. points out that his thiouram disulfide method (*C. A.* 6, 2928) affords the smoothest means of transforming a primary amine with a non-aromatic  $NH_2$  group into the corresponding mustard oil. S. and R.'s cyclohexyl mustard oil can be obtained almost quant. in a few min. from hexahydroaniline.

CHAS. A. ROUILLE

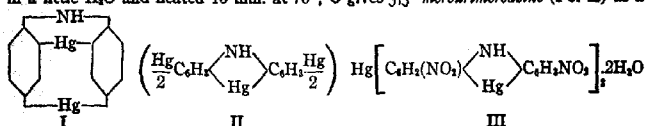
Syntheses of sym-xylidine. HERBERT L. HALLER and ELLIOT Q. ADAMS with EDGAR T. WHERRY. *J. Am. Chem. Soc.* 42, 1840–2 (1920).—In order to obtain sym-xylidine (A) absolutely free from its isomers 50 g. mesitylene was boiled 22 hrs. with

80 cc.  $\text{HNO}_3$  (d. 1.47) and 240 cc.  $\text{H}_2\text{O}$ , giving 9.7 g. mesitylenic acid; this was converted through the chloride into the amide, 40 g. of which with  $\text{NaOBr}$  gave 19 g. A, whose Ac deriv. m. 139.6–40.2°. The synthesis from *m*,4-xylydine (B) (Willgerodt and Schmierer, *Ber.* 38, 1472(1905)), owing to the consistently higher yields, is more satisfactory when considerable amts. of A are desired. It has been found possible to eliminate one operation in this method. The B was obtained from com. xylydine by letting it stand in glacial  $\text{AcOH}$  24–48 hrs; the resulting acetate was acetylated with  $\text{Ac}_2\text{O}$ , the Ac deriv. converted with red fuming  $\text{HNO}_3$  (d. 1.55–1.57) at 0–5° into the acetonitroxylide, 50 g. of this heated 15 min. at 125° with 50 g.  $\text{H}_2\text{SO}_4$  (d. 1.84), poured into 300 g. of 95% alc. and 150 g. concd.  $\text{H}_2\text{SO}_4$  and diazotized at 5° with 30 g.  $\text{NaNO}_2$  in the presence of Cu powder, giving 35 g. nitroxylene, which, heated 1 hr. on the  $\text{H}_2\text{O}$  bath with 150 g.  $\text{SnCl}_2$  and 200 g.  $\text{HCl}$  (d. 1.19), gave 15 g. A. The Ac deriv. seps. from alc. in plates, apparently of the monoclinic system, with the clinopinacoid prominent; refractive indices (D):  $\alpha$  1.45,  $\beta$  indeterminate,  $\gamma$  1.69. CHAS. A. ROULLER.

Crystalline chlorotetraacetylfructose and related derivatives. D. H. BRAUN. *J. Am. Chem. Soc.* 42, 1846–54(1920).—A cryst. chlorotetraacetylfructose (probably the  $\alpha$ -form) is obtained in 6-g. yield from 10 g. cryst.  $\beta$ -pentaacetylfructose in 14 cc.  $\text{CHCl}_3$  with 2.5 g.  $\text{AlCl}_3$  and 6.5 g.  $\text{PCl}_5$  in 40-g. yield from 60 g.  $\beta$ -tetraacetylfructose in 180 cc.  $\text{CHCl}_3$  with 15 g.  $\text{AlCl}_3$  and 38 g.  $\text{PCl}_5$  and in small yield from 7 g. of the pentaacetate in 8 cc.  $\text{AcOH}$  satd. with  $\text{HCl}$  dild. to 25 cc. with  $\text{AcOH}$ , allowed to stand 0.5 hr. and poured into cold  $\text{CHCl}_3$ ; it decomps. in 1 day in an open dish and almost as rapidly in a desiccator, but can be kept for some weeks in  $\text{C}_6\text{H}_6$  soln. in a tightly closed bottle in the ice chest; mol. wt. in freezing  $\text{C}_6\text{H}_6$  342–62,  $[\alpha]_D^{20}$  –160.9° ( $\text{CHCl}_3$ ); it m. 83°, has a bitter taste, is converted almost quant. into tetraacetylfructose by shaking 10 g. in  $\text{Et}_2\text{O}$  with 10 g.  $\text{Ag}_2\text{O}$  and 5 cc.  $\text{H}_2\text{O}$  for 1 day. If 60 g.  $\beta$ -tetraacetylfructose in 180 cc.  $\text{CHCl}_3$  is treated with 40 g.  $\text{PCl}_5$  alone (without  $\text{AlCl}_3$ ), the product (35 g.) is different from the above and is probably the  $\beta$ -isomer; it is stable, mol. wt. in freezing  $\text{C}_6\text{H}_6$  345–61,  $[\alpha]_D^{20}$  45.3° ( $\text{CHCl}_3$ ), m. 108°, is tasteless or only very slightly bitter; the Cl is much more firmly attached than in the  $\alpha$ -isomer.  $\beta$ -Ethyltetraacetylfructose (16 g. from 20 g. fructose tetraacetate, 75 g.  $\text{Ag}_2\text{O}$  and 128 g.  $\text{EtI}$ ), m. 83°,  $[\alpha]_D^{20}$  –127.6° ( $\text{CHCl}_3$ ), hydrolyzed by  $\text{Ba}(\text{OH})_2$  to  $\beta$ -ethyl fructoside, m. 151°,  $[\alpha]_D^{20}$  –155.3° ( $\text{H}_2\text{O}$ ), is slightly bitter or tasteless, does not reduce boiling Fehling soln.  $\alpha$ -Pentaacetylfructose (5 g.) heated 1 hr. at 106° in 500 cc.  $\text{H}_2\text{O}$ , neutralized and extd. with  $\text{CHCl}_3$ , yields 0.28 g.  $\beta$ -tetraacetylfructose, so with the latter as intermediate product, the  $\alpha$ -pentaacetate can now be converted into the  $\beta$ -form. Attempts to prep.  $\alpha$ -Me fructosides failed; the sublimation method yields the cryst.  $\beta$ -fructoside. C. A. R.

Aromatic mercuri-organic derivatives. MORRIS S. KHARASCH AND JEAN F. PICCARD. *J. Am. Chem. Soc.* 42, 1855–64(1920).—The present paper is a report on some preliminary work on the prepn. of a 6-membered heterocyclic compd. with Hg in place of a non-metal (N in an azine ring). 3,4-Br(ON) $\text{C}_6\text{H}_3\text{NMe}_2\text{HCl}$  (A) was obtained by treating 75 g. *m*-Br $\text{C}_6\text{H}_4\text{NH}_2$  in 900 cc.  $\text{H}_2\text{O}$  suspension at 80° with 300 g.  $\text{Na}_2\text{CO}_3$  and 300 g.  $\text{Me}_2\text{SO}_4$  distg. with steam and treating the oil (75 g.) in  $\text{HCl}$  at 0° with  $\text{NaNO}_2$ ;  $\text{SnCl}_2\text{-HCl}$  reduces it to *m*-bromodimethyl-*p*-phenylenediamine (B), almost colorless needles, m. 47.5°, darkens in the light and air, gives an intense blue color when satd. in  $\text{H}_2\text{O}$  with  $\text{H}_2\text{S}$  and treated with  $\text{FeCl}_3$ ; dihydrochloride. When 2.15 g. B, 2 g. *m*-Br $\text{C}_6\text{H}_4\text{NMe}_2$ , 2 g. concd.  $\text{HCl}$  and 50 cc.  $\text{H}_2\text{O}$  at –2° are treated with 2 g.  $\text{K}_2\text{Cr}_2\text{O}_7$  in 0.6 g.  $\text{AcOH}$  and 20 cc.  $\text{H}_2\text{O}$ , then dild. with enough  $\text{H}_2\text{O}$  to redissolve the dye which seps., heated to 40–50° and treated with 5 g.  $\text{ZnCl}_2$  there is obtained a good yield of *o*-dibromo-Binschedler's green-zinc chloride,  $[\text{Br}(\text{Me}_2\text{N})\text{C}_6\text{H}_3\text{N}:\text{C}_6\text{H}_4\text{Br}:\text{NMe}_2\text{Cl}]_2\text{ZnCl}_2$ , as a dark amorphous powder, slightly sol. with intense green color in  $\text{H}_2\text{O}$ , gives with  $\text{HgCl}_2$  in  $\text{H}_2\text{O}$  at 40° the mercuric chloride compound. Extn. with  $\text{Et}_2\text{O}$  of

the soln. from which A seps. yields 10 g. *m*-bromophenylmethylnitrosamine, fine yellowish needles, m. 49°, is very stable when pure; 4 g. in 8 g. Et<sub>2</sub>O allowed to stand 2 days with 16 g. alc. satd. at 10° with HCl gave the hydrochloride, turns yellow 140°, m. 168° (apparent decomp.), of *p*-nitroso-*m*-bromomethylaniline, which is green, has no definite m. p., darkens 100°, explodes 124° and dissolves in Et<sub>2</sub>O with yellowish green in thin and green color in thick layers. *p*-Nitrosophenol-*o*-mercuric chloride, 2,4-ClHg(ON)-C<sub>6</sub>H<sub>3</sub>OH, from ClHgC<sub>6</sub>H<sub>4</sub>OH in NaOH, NaNO<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> crystals from xylene, light brown needles from anisole, partially dissolves in Et<sub>2</sub>O with intense green color; with NH<sub>4</sub>Cl, AcONH<sub>4</sub> and (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> on the H<sub>2</sub>O bath it gives *p*-nitrosoaniline-*o*-mercuric chloride, crystals from anisole, sol. in C<sub>6</sub>H<sub>6</sub> and Et<sub>2</sub>O with green, in alc. with reddish yellow color. Diphenylaminotetramercuric chloride (C), NH(C<sub>6</sub>H<sub>5</sub>(HgCl)<sub>2</sub>)<sub>2</sub>, from 6.5 g. NHPH<sub>2</sub> and 2.7 g. HgCl<sub>2</sub> heated until the latter seems to dissolve, is a light yellow amorphous substance, does not m. 260°, seemingly decomp. when boiled with C<sub>6</sub>H<sub>5</sub>N; from analogy, the HgCl groups are in the 2,4-positions. Treated in alc. with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> in a little H<sub>2</sub>O and heated 15 min. at 70°, C gives 3,3'-mercurimercasine (I or II) as a



gray substance resembling finely pptd. Hg, yields Hg *in vacuo* at 100°, unstable towards HNO<sub>3</sub>, yielding a product sol. in NaOH with red color. *o,o'*, *p*-Trichloromercuri-*o,o'*, *p*-dinirodiphenylamine, from 5 g. C and 20 g. of a mixt. of 2 parts HNO<sub>3</sub> (d. 1.4), 1 part H<sub>2</sub>SO<sub>4</sub> and 1 part H<sub>2</sub>O carefully warmed 2 min., is a light brown substance partly sol. in NaOH with red color, converted by Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> into mercury bis-3-[*o,o'*, *p*-dinirodiphenylamine] (III), a light brown substance, colors NaOH light red, loses Hg *in vacuo* at 100°.

CHAS. A. ROULLER

**Tautomerism in the triarylcarbinol series: diphenyl-4-hydroxy-1-naphthylcarbinol and diphenyl-2-hydroxy-1-naphthylcarbinol.** M. GOMBERG AND F. W. SULLIVAN. Jr. *J. Am. Chem. Soc.* 42, 1864-70(1920).—There are at present on record a number of hydroxytriarylcarbinols of which all of the 3 components that constitute the tautomeric equil. (colorless benzenoid, colored quinonoid and fuchson) are known and a still larger number for which the stable existence of only 2 components (colorless carbinol and fuchson) is definitely established, but there are no instances described in which the stable existence of a colored carbinol and the corresponding fuchson has been observed, although quite a number of fuchsons have been prepd. Believing that this might be due in many cases to the colored carbinol having been overlooked, G. and S. have carefully studied *p*-naphthofuchson (A) and *o*-1-naphthofuchson-2 (B) and have been able to isolate carbinols corresponding to them, the quinonoid in the former and the benzenoid tautomer only in the latter case. The statement that in the case of A only the quinonoid carbinol was obtained requires modification; when apparently only one carbinol is obtained and that is colored and amorphous, it becomes difficult to say unequivocally whether it is the quinonoid carbinol alone or admixed with some of the benzenoid form. A was obtained in 7.8-g. yield from 6 g. Ph<sub>2</sub>CCl<sub>2</sub> and 7.5 g.  $\alpha$ -naphthol allowed to stand overnight protected from atm. moisture; it is orange-yellow, m. 179° and gives a violet color with H<sub>2</sub>SO<sub>4</sub>; with the calcd. amt. of Br in AcOH it gives 70% of 3-bromo-*p*-naphthofuchson, reddish brown crystals, m. 176°, gives an intense blue color with H<sub>2</sub>SO<sub>4</sub>; 3-chloro compound, similarly obtained, forms bright red crystals, m. 161°. B (4 g. from 6 g. Ph<sub>2</sub>CCl<sub>2</sub>, 4.4 g.  $\beta$ -naphthol and 4.1 g. AlCl<sub>3</sub> in 50 cc. CS<sub>2</sub> allowed to stand overnight and boiled 1 hr.), orange-red crystals from AcOH, m. 198°, gives a green color with H<sub>2</sub>SO<sub>4</sub>. A in alc. NaOH treated with NH<sub>4</sub>Cl yields a volumi-

ous ppt. of 4-hydroxy-1-naphthylidiphenylcarbinol, amorphous yellow powder when dry which cannot be recrystd., as it is changed into A in all solvents. 3-Bromo-4-hydroxy-1-naphthylidiphenylcarbinol, similarly obtained, is a canary-yellow powder losing 1 mol.  $\text{H}_2\text{O}$  at  $120^\circ$ ; 3-chloro compound. All these carbinols give the same color as the fuchsones with  $\text{H}_2\text{SO}_4$ ; they probably consist largely, if not entirely, of the quinonoid forms. 2-Hydroxy-1-naphthylidiphenylcarbinol, prep'd. in the same way, is quite stable and seps. from  $\text{C}_6\text{H}_6$  and petr. ether in pure white plates which lose  $\text{H}_2\text{O}$  on heating, become red and melt slightly below the true m. p. of B; all efforts to convert it into the quinonoid carbinol resulted in the formation of B; from alc. containing a few drops of  $\text{NH}_4\text{OH}$  it seps. on standing in feathery crystals, presumably of the  $\text{NH}_4$  salt (also obtained by shaking B with alc. satd. with  $\text{NH}_4$ ); in an open tube this salt changes into B on heating, but in a closed capillary m.  $171^\circ$ . A absorbs somewhat more than 2 mols.  $\text{HCl}$  in 3 hrs. in a  $\text{HCl}$  atm. and turns to a black viscous mass which *in vacuo* loses about  $\frac{1}{2}$  of its  $\text{HCl}$  in 24 hrs. and the rest in 4-5 days. B also absorbs  $\text{HCl}$  and gives it up much more slowly than A *in vacuo*, 23 days being required for loss of 80% of the absorbed  $\text{HCl}$ . Mono-*p*-hydroxydiphenyl- $\alpha$ -naphthylcarbinol. M. GOMBERG AND N. A. LANGE. *Ibid* 1879-83.—As shown above, the  $\text{C}_{10}\text{H}_8$  nucleus in monohydroxytriarylcarbinols exerts a much more pronounced tautomerizing influence than does the  $\text{C}_6\text{H}_5$  nucleus when each carries a  $\text{HO}$  group, for, while *p*- $\text{HOC}_6\text{H}_4\text{CPh}_2\text{OH}$  is stable both in its benzenoid and quinonoid forms, the benzenoid form of *p*-hydroxynaphthylidiphenylcarbinol spontaneously changes into the quinonoid form. In the present paper it is shown that even when the  $\text{HO}$  group is not on the  $\text{C}_{10}\text{H}_8$  nucleus, the latter still exerts a noticeably greater tautomerizing influence than a  $\text{C}_6\text{H}_5$  nucleus.  $\alpha$ -Naphthyl phenyl ketone chloride, from  $\text{C}_{10}\text{H}_7\text{COPh}$ , and the calcd. amt. of  $\text{PCl}_5$  at  $100^\circ$ , needles from petr. ether, begins to soften about  $86^\circ$ , m.  $92^\circ$  (yield, 60%); 7 g. with 6 g.  $\text{PhOH}$  in boiling  $\text{C}_6\text{H}_6$  gives 80-5% of phenyl- $\alpha$ -naphthylidiphenoxymethane, crystals from  $\text{AmOH}$ , m.  $169-70^\circ$ , while 8 g. allowed to stand 12 hrs. with 13.5 g.  $\text{PhOH}$  and then heated 1 hr. at about  $50^\circ$  yields 85-90% of the fuchsones  $\alpha$ -naphthylquinomethane, yellow crystals from dil. alc., m.  $169-70^\circ$ , sol. in  $\text{H}_2\text{SO}_4$  with deep red color; finally 4 g. of the ketone chloride heated 24 hrs. at  $110^\circ$  with 30 g.  $\text{PhOH}$  satd. with dry  $\text{HCl}$  gives 15-20% of 4,4'-dihydroxytriphenyl- $\alpha$ -naphthylmethane, microneedles from  $\text{AcOH}$ , softens  $260^\circ$ , m.  $209-10^\circ$  (decompn.). The fuchsones in a little alc. boiled with *N*  $\text{NaOH}$  until the soln. changes from red-yellow to very light yellow, dild. with an equal vol. of  $\text{H}_2\text{O}$  and treated with  $\text{NH}_4\text{Cl}$  yields diphenyl- $\alpha$ -naphthylcarbinol (probably chiefly the quinonoid form) as a light yellow amorphous substance which, when deposited from dil.  $\text{AcOH}$ , begins to soften and turn red at  $70^\circ$  while a sample pptd. from alk. soln. and probably containing some of the benzenoid form, begins to soften and turn red at  $87^\circ$ . CHAS. A. ROUILLER

Friedel and Crafts' reaction—nitrophthalic anhydrides and acetylaminophthalic anhydrides with benzene and aluminium chloride. WALTER A. LAWRENCE. *J. Am. Chem. Soc.* 42, 1871-9 (1920).—3- and 4-Nitrophthalic acids are obtained in 42 and 47% yield, resp., by adding 125 g. each of  $\text{HNO}_3$  (d. 1.5) and  $\text{H}_2\text{SO}_4$  (d. 1.834) to 50 g.  $\text{C}_6\text{H}_4(\text{CO})_2\text{O}$  and keeping the mixt. 3 hrs. at about  $100^\circ$ . 3-Nitrophthalic anhydride (A), m.  $164^\circ$ , is obtained in 89% yield from the acid heated 3-4 hrs. at  $215-7^\circ$ ; 4- $\text{NO}_2$  isomer (B), from the acid heated 3 hrs. at  $165-8^\circ$ , m.  $114^\circ$  (yield, 80%). The 3- $\text{NO}_2$  acid in alc. satd. with  $\text{HCl}$  and boiled 1 hr. gives 3,2- $\text{O}_2\text{N}(\text{EtO}_2\text{C})\text{C}_6\text{H}_3\text{CO}_2\text{H}$ , m.  $110^\circ$ , while the anhydride boiled 5 hrs. in alc. yields the 2,6-isomer, m.  $156-7^\circ$ . 3-Acetylaminophthalic anhydride (C), m.  $185-6^\circ$ , is obtained in 55% yield by reducing 40 g. of the 3- $\text{NO}_2$  acid with 150 g.  $\text{SnCl}_2$ , 250 cc. concd.  $\text{HCl}$  and 50 cc.  $\text{H}_2\text{O}$  at about  $22^\circ$  and boiling the resulting  $\text{ClH}_3\text{H}_2\text{NC}_6\text{H}_3(\text{CO}_2\text{H})_2$  5 min. with  $\text{Ac}_2\text{O}$ . 4-AcNH isomer (D), obtained in 10% yield by converting the acid  $\text{NH}_4$  salt of the 4- $\text{NO}_2$  acid into the imide, reducing with  $\text{SnCl}_2$ , converting into the  $\text{NH}_4$  salt of the  $\text{NH}_2$  acid by boil-

ing with  $\text{NH}_4\text{OH}$ , setting free the acid with  $\text{HCl}$  and boiling a few min. with  $\text{Ac}_2\text{O}$ , m.  $203-6^\circ$ . From 6.5 g. A in 100 cc.  $\text{C}_6\text{H}_6$  and 9 g.  $\text{AlCl}_3$  heated 4-5 hrs. on the  $\text{H}_2\text{O}$  bath are obtained 3.8 g. of the 3- $\text{NO}_2$  acid, 1.7 g. of a black resinous mass insol. in  $\text{NaOH}$  and the usual organic solvents, 0.7 g. 6-benzoyl-2-nitrobenzoic acid (E), insol. in  $\text{AcOH}$ , and 0.6 g. 2-benzoyl-3-nitrobenzoic acid (F), sol. in  $\text{AcOH}$ . E m.  $217-21^\circ$  (decompn.), yields  $\text{BzOH}$  and  $o\text{-O}_2\text{NC}_6\text{H}_4\text{CO}_2\text{H}$  on fusion with  $\text{KOH}$  and can be synthesized (45% yield) from 3,2- $\text{O}_2\text{N}(\text{EtO}_2\text{C})\text{C}_6\text{H}_4\text{CO}_2\text{H}$  converted by  $\text{SOCl}_2$  at  $60-70^\circ$  into the chloride, m.  $76-7^\circ$ , treated in  $\text{C}_6\text{H}_6$  with  $\text{AlCl}_3$  and sapond. with alc.  $\text{KOH}$ . F softens  $140^\circ$ , m.  $158-61^\circ$  (decompn.), is hydrolyzed by  $\text{KOH}$  to  $\text{BzOH}$  and  $m\text{-O}_2\text{NC}_6\text{H}_4\text{CO}_2\text{H}$ , and can be synthesized from 2,6- $\text{O}_2\text{N}(\text{EtO}_2\text{C})\text{C}_6\text{H}_4\text{CO}_2\text{H}$  through the chloride, yellowish oil, decomp. above  $100^\circ$ . B (6.5 g.) with 9 g.  $\text{AlCl}_3$  in 100 cc.  $\text{C}_6\text{H}_6$ , gives 50% of the 4- $\text{NO}_2$  acid, 0.8 g. 6,3-Bz( $\text{O}_2\text{N}$ ) $\text{C}_6\text{H}_3\text{CO}_2\text{H}$ , m.  $212^\circ$ , and 0.5 g. of the 2,4-isomer, m.  $164-5^\circ$ . From 6.8 g. C and 9 g.  $\text{AlCl}_3$  in 50 cc.  $\text{C}_6\text{H}_6$  are obtained 51% of 2-benzoyl-3-aminobenzoic acid, m.  $193-4^\circ$  (synthesized by reduction of F with  $\text{NH}_4\text{OH}\cdot\text{FeSO}_4$ ), a small amt. of the 6,2-isomer, m.  $159-60^\circ$  (also synthesized from E), and 11% of di-phenyl-3-(or 6)-aminophthalide, orange amorphous powder m.  $86-8^\circ$  (this product is obtained in 31% yield when the C,  $\text{AlCl}_3$  and  $\text{C}_6\text{H}_6$  are heated 7 hrs., treated with 10 cc.  $\text{Ac}_2\text{O}$  and heated another 7 hrs.). D with  $\text{AlCl}_3$  and  $\text{C}_6\text{H}_6$  gives 6,3-Bz( $\text{H}_2\text{N}$ ) $\text{C}_6\text{H}_3\text{CO}_2\text{H}$ , m.  $193-4^\circ$ , and the 2,4-isomer, m.  $195-6^\circ$  (decompn.). CHAS. A. ROUILLER.

Preparation of benzyl esters and other benzyl derivatives from benzyl chloride. M. GOMBERG and C. C. BUCHLER. *J. Am. Chem. Soc.* 42, 2059-72 (1920).—Good yields of benzyl esters have been obtained in a number of cases from  $\text{PhCH}_2\text{Cl}$  and the Na salt of the acid in  $\text{H}_2\text{O}$ . From  $\text{PhCH}_2\text{Cl}$  heated 6 hrs. at  $200-30^\circ$  with a slight excess of dry  $\text{NaOBz}$  was obtained 18.8%  $\text{BzOCH}_2\text{Ph}$ ; with a few mg. of Cu bronze as catalyst, 60.3% after 3.5 hrs. at  $190-300^\circ$ ; with  $\text{NiCl}_2$  as catalyst, 26.4% after 5 hrs. at  $200-10^\circ$ . The product so obtained contains some  $\text{PhCH}_2\text{Cl}$ , which can be removed only by subsequent treatment with dil. alkali, and a small amt. of a high boiling product resulting from the decompn. of the  $\text{PhCH}_2\text{Cl}$ . In  $\text{H}_2\text{O}$ , the best yield (42 g. or 79%) was obtained from 110 g.  $\text{NaOBz}$  in 150-200 cc.  $\text{H}_2\text{O}$  and 32 g.  $\text{PhCH}_2\text{Cl}$  heated 4-5 hrs. at  $115^\circ$  (70 g.  $\text{BzOH}$  is recovered); the product, after 1 distn. *in vacuo*, is free of  $\text{Cl}$ . Anhydrous  $\text{NaOAc}$  with Cu as catalyst gives 30-40%  $\text{AcOCH}_2\text{Ph}$  after several hrs., but an 82.6% yield is obtained from 53 g. of the salt heated 8 hrs. with 32 g.  $\text{PhCH}_2\text{Cl}$  in  $\text{H}_2\text{O}$  at  $115^\circ$ . In the same way were prepd. (yields in parentheses): the butyrate (83%); lactate,  $b_{20} 150-6^\circ$  (55%); phenylacetate (75%); cinnamate (83%); salicylate (40-5%); succinate (43%); oleate (about 45%). Formic, oxalic and phthalic acids gave no ester. With  $\text{PhOH}$ ,  $\text{PhCH}_2\text{Cl}$  in the absence of solvents reacts at  $150-80^\circ$  with formation of  $\text{PhCH}_2\text{C}_6\text{H}_4\text{OH}$  (30% after 3 hrs.); in the presence of Cu the yields are better (40% after 3 hrs. at  $115-20^\circ$ ); in  $\text{H}_2\text{O}$ , 61%  $\text{PhCH}_2\text{OPh}$  is obtained after 2 hrs. at  $110^\circ$ .  $\alpha$ - and  $\beta\text{-C}_{10}\text{H}_7\text{OH}$  give 20 and 50%, resp., of the corresponding  $\text{PhCH}_2\text{-C}_{10}\text{H}_7\text{OH}$  after 2 hrs. in  $\text{H}_2\text{O}$  at  $105-10^\circ$ . From 5 mols.  $(\text{NH}_4)_2\text{CO}_3\cdot\text{H}_2\text{O}$  heated 5-6 hrs. at  $85-90^\circ$  were obtained 16% mono-, 20% di- and 44% tribenzylamine.  $\text{PhNH}_2$  (23 g.) heated 4 hrs. in  $\text{H}_2\text{O}$  at  $95-100^\circ$  with 32 g.  $\text{PhCH}_2\text{Cl}$  gives 56 and 21% mono- and dibenzylanilines, resp., while with 64 g.  $\text{PhCH}_2\text{Cl}$  the yields are 13 and 60%; 27 g.  $\text{PhNHMe}$  with 32 g.  $\text{PhCH}_2\text{Cl}$  yields 90%  $\text{PhCH}_2\text{NPhMe}$ , and 21.5 g.  $p$ - and  $m\text{-Me-C}_6\text{H}_4\text{NH}_2$  with 50 g.  $\text{PhCH}_2\text{Cl}$  give 76.6 and 71.4 g., resp., of  $p$ - and  $m\text{-MeC}_6\text{H}_4\text{N}(\text{CH}_2\text{Ph})_2$ .  $\text{PhCH}_2\text{CN}$  can be obtained in as high yield as 70% from 24 g.  $\text{NaCN}$  in 100 cc.  $\text{H}_2\text{O}$  and 32 g.  $\text{PhCH}_2\text{Cl}$  in 1 hr. at  $105^\circ$ . An 80% yield of  $\text{PhCH}_2\text{OH}$  is obtained from 32 g.  $\text{PhCH}_2\text{Cl}$  heated 1 hr. at  $120-5^\circ$  with 20 g.  $\text{NaOH}$ . C. A. R.

Interaction of carbon disulfide with ammonium carbonate. FRANCOIS A. GILFILLAN. *J. Am. Chem. Soc.* 42, 2072-80 (1920); cf. Inghilleri, *C. A.* 5, 638.—I's work was repeated, the amt. of  $\text{NH}_4\text{SCN}$  and  $\text{CS}(\text{NH}_2)_2$  formed being detd. quant. and a study made of the influence of the different factors concerned in the reaction



equil.  $\text{NH}_4\text{SCN}$  cannot be detd. accurately by indirect titration with excess of  $\text{AgNO}_3$  in the presence of more than 15%  $\text{CS}(\text{NH}_2)_2$ , but it can be detd. by direct titration with  $\text{AgNO}_3$  in the presence of up to 40%  $\text{CS}(\text{NH}_2)_2$ ; higher concns. of the latter interfere with the titration, apparently owing to the soly. of  $\text{AgSCN}$  in the  $\text{CS}(\text{NH}_2)_2$  soln. with formation of a complex ion. The  $\text{CS}(\text{NH}_2)_2$  can be detd. by titration with I if its concn. does not exceed 5 mg. per 100 cc. The method followed in heating the  $\text{CS}_2$  and  $(\text{NH}_4)_2\text{CO}_3$  was in general the same as I.'s (6 hrs. at  $100-10^\circ$ , 6 hrs. at  $125-30^\circ$ , and 2 hrs. at  $160^\circ$ , the pressure being released after each period of heating). Contrary to his statement, much  $\text{H}_2\text{S}$  was always produced at  $100^\circ$ . The yield of total  $\text{NH}_4\text{SCN}$  and  $\text{CS}(\text{NH}_2)_2$ , based on  $\text{CS}_2$  in the presence of a 5% excess of  $\text{NH}_3$ , was 77.4-90.9%. The highest % of  $\text{CS}(\text{NH}_2)_2$  in the equil. mixt. found was 10.9% and ranged as low as 1.2%, the lowest % being found in those tubes which had been allowed to stand some time at room temp. after heating at  $160^\circ$  in the presence of the  $\text{H}_2\text{S}$  and  $\text{CO}_2$  generated at that temp.; the total yield of  $\text{NH}_4\text{SCN}$  and  $\text{CS}(\text{NH}_2)_2$  was also lower in these tubes than in those opened immediately after cooling.

CHAS. A. ROUILLER

Reaction between selenium monochloride and ethylene (HATH, SEMON) 6. Action of methyl and ethyl sulfates on alkali phosphates (BAILLY) 6. Chemistry of the polysaccharides (HERZFELD, KLINGER) 11A. Influence of electrolytic dissociation on the distillation in steam of the volatile fatty acids. Application of the method of distillation in steam (REILLY HICKINBOTTOM) 2. Manufacture of the higher phenols ("LATEX") 21. Fatty acids from hydrocarbons (Brit. pat. 147,905) 22.

**Acetaldehyde.** H. L. BENDER. U. S. 1,355,299, Oct. 12. The process has for its object the manuf. of AcH by passing steam and  $\text{C}_2\text{H}_2$  over a catalyst at a relatively low temp. such as to avoid decompn. of the AcH formed and poisoning of the catalyzer by impurities present in the  $\text{C}_2\text{H}_2$ . A suitable catalyst is activated charcoal formed from nut shells and fruit pits which may be used alone or as a carrier for other substances such as Hg or its salts or oxide or salts or oxides of Zn, Cu, Mo, Fe, Ni, Sn, Al or Pb. Silica or asbestos or "sil-o-cel" also may be used with Hg oxide or other substances as the catalyst. With a catalyst of charcoal and Hg oxide, the yield of AcH increased with decreasing temp. from 30% at  $190^\circ$  to 90% at  $103^\circ$ . Activated charcoal alone gave an increasing yield as the temp. was maintained higher, rising to 25% at a temp. of  $350^\circ$  and then decreasing as the temp. increased. Activated charcoal impregnated with Hg gave a yield increasing to 30% at  $250^\circ$  and then decreasing at higher temps. Activated charcoal impregnated with Cu oxide gave a yield increasing from 5% at  $220^\circ$ , 15% at  $250^\circ$ , 30% at  $280^\circ$  to 90% at  $305^\circ$ . Silica impregnated with Hg oxide gave a yield increasing up to 8% at  $140^\circ$  and 20% at  $225^\circ$ . Activated charcoal impregnated with  $\text{HgSO}_4$  gave a yield increasing from 10% at  $130^\circ$  to 36% at  $250^\circ$ . Activated charcoal impregnated with ZnO gave a yield increasing from 10% at  $200^\circ$  to 85% at  $310^\circ$ . Activated charcoal impregnated with Mo oxide gave a yield increasing from 2% at  $220^\circ$  to 70% at  $340^\circ$ . In each of these tests 1% or less of the impregnating substance was used with the charcoal. After loss of activity, the catalyst may be regenerated by heating with air.

**Hydrogenating naphthalene.** G. SCHROETER. Brit. 147,747, July 8, 1920. Addition to 147,580 (C. A. 14, 3675). Commercial naphthalene is dissolved in an org. solvent and after preliminary purification, and with or without removal of the solvent, is treated with H in the presence of a catalyst. Suitable purifying materials are finely divided or readily fusible metals or metal alloys, or metal compds. in which the metal is loosely bound to a non-acid radical, either alone or mixed with finely divided or porous materials such as fuller's earth, all as described in the principal patent. In an example, commercial naphthalene is dissolved in tetrahydronaphthalene and treated

with metallic Na at a temp. of 150–200°; after distn. from the residue the purified naphthalene in soln. is treated with H in the presence of a catalyst.

**Ethylene chloride.** T. GOLDSCHMIDT AKT.-GAS. Brit. 147,909, July 9, 1920. The  $C_2H_4$  content of coal-gas, or other gases produced by the distn. of carbonaceous matter, such as lignite, oils, etc., is utilized for the production of  $C_2H_4Cl_2$  by treating the gases with Cl in the presence of a suitable catalyst, and sepg. the  $C_2H_4Cl_2$  from the treated gas by liquefaction by compressing, cooling, etc.

**Ethylene chloride; ethylene.** T. GOLDSCHMIDT AKT.-GAS. Brit. 147,908, July 9, 1920.  $C_2H_4Cl_2$  is obtained by treating  $C_2H_4$  (prepd. by passing alc. vapor over hot  $Al_2O_3$ , etc.) with chlorine in the presence of a catalyst, such as Fe, Cu, or Sb chlorides; the ethylene should be used in large excess, and a temp. of 30–120° maintained. The catalyst is preferably disposed in or upon a cooling device through which the gas flows. The  $C_2H_4Cl_2$  produced is sepd. by cooling the treated gas and the unused  $C_2H_4$  returned to the gas-holder for re-use. The  $C_2H_4$  used is freed from  $H_2O$  and alc. vapor by cooling; both gases should be dry and free from S.

**Glycols, etc.** O. MATTER. Brit. 147,906, July 9, 1920. Di- or polyhydric alcs. are obtained by heating the corresponding hydrocarbons with bicarbonates of the alkalis or alk.-earths under pressure in the presence of  $H_2O$ . Instead of the bicarbonates, mixts. of the bicarbonates and carbonates may be used; catalysts such as Cu may be added. According to an example, glycol is obtained by heating and stirring  $C_2H_5Cl_2$  with aq.  $NaHCO_3$  and Cu under pressure; the product is worked up by neutralizing with acid and fractional distn. In order to prevent undue rise of pressure in the autoclaves, it is advisable to remove therefrom at intervals  $CO_2$  and  $C_2H_5Cl_2$  and lead these to a second vessel.

**Glycols, etc.** O. MATTER. Brit. 147,907, July 9, 1920. Addition to 147,906 (above). The process for the production of glycol, etc., described in the principal patent is effected in the presence of  $H_2O$  in large excess compared with the chlorohydrocarbon. This may be effected by introducing the chlorohydrocarbon gradually in small quantities into the liquid contained in the autoclave. In place of using alkali or alk.-earth bicarbonates, the carbonates may be employed, these being bicarbonated by the  $CO_2$  produced. Catalysts such as Cu may be added.

**Sulfonic acids.** F. C. SUTTON. Brit. 147,987, Feb. 20, 1919. Aromatic hydrocarbons, e. g., benzene, are sulfonated by atomizing the liquid hydrocarbon and  $H_2SO_4$  through two oppositely disposed jets in a suitable reaction chamber. Benzene and  $H_2SO_4$  are forced from tanks up pipes to atomizing nozzles near the top of a steam-jacketed reaction tower. The product of the reaction runs down over baffles to a receiver. Benzene vaporized in the process is condensed by a reflux condenser and is led to the benzene supply tank. Cf. 139,234 and 140,007. A suitable construction is specified.

**Oxalic acid.** G. H. GONTARD and A. KELLER. U. S. 1,356,137, Oct. 19. Oxalic acid is prepd. by treating 100 parts by wt. of sucrose with 320 parts of 100%  $H_2SO_4$ , 300 parts  $HNO_3$  of 100% strength, and 380 parts of  $H_2O$  in the presence of a catalyst such as  $V_2O_5$  or salts of Mo or Mn.

**Chlorobenzoylbenzoic acid.** J. M. WEISS. U. S. 1,355,100, Oct. 5. Ground  $C_6H_4(CO)O$  100 is mixed with coarse freshly sublimed  $AlCl_3$  180 parts and the mixt. is gradually added, with agitation, to 1000 parts of  $PhCl$  at 120°.  $HCl$  evolved is recovered. After cooling, water or ice is added to the mixt. followed by the addition of 80–90 parts of 60%  $H_2SO_4$ . Excess  $PhCl$  is then removed by steam distn. The crude  $ClC_6H_4COC_6H_4CO_2H$  (A) is filtered from the mass after cooling. The addition of  $H_2SO_4$  and concn. of the filtrate will produce about 230 parts of  $Al_2(SO_4)_3$ . If phthalic acid is present from the first reaction, it may be recovered either by filtering the hot reaction mixt. after the  $PhCl$  is removed by steam distn. (in which case the phthalic acid will

sep. from the cooled filtrate and the A will remain as the undissolved residue), or the mixt. from the steam distn. may be cooled and then filtered, in which case the phthalic acid may be extd. from the crude A with hot  $H_2O$ . \*

**Silver salts of amino acids.** H. R. NAPP. Brit. 148,074, Sept. 23, 1919. Complex Ag salts of aliphatic  $\alpha$ -amino acids are prepd. by treating an excess of the amino acid, e. g., glycine or alanine, with  $Ag_2O$  or with inorg. or org. Ag salts, e. g., the nitrate, sulfate, acetate, or salicylate of Ag. In the case of glycine, the normal Ag salt may first be prepd. and then treated with excess of glycine to produce the complex salt. With glycine, too, the temp. must be kept below  $0^\circ$ .

**Oxidizing organic aromatic compounds.** C. E. ANDREWS. Can. 204,780, Oct. 12, 1920. Org. aromatic compds. are oxidized by heating with  $H_2SO_4$  and a catalyst containing V or Mo; e. g., naphthalene is heated with  $H_2SO_4$  in the presence of a V catalyst to produce phthalic anhydride.

**Alkylaminoanthraquinones; alkylarylamines.** F. W. ATACK and W. N. HAWORTH. Brit. 147,964, Jan. 22, 1919. Alkylaminoanthraquinones are obtained by heating 1- or 2-aminoanthraquinone with dialkyl sulfates in presence of a mild alk. reagent, such as  $Na_2CO_3$  or  $MgO$ , and a solvent of high b. p., such as nitrobenzene or tetrachloroethane. Examples are given of the prepn. of 1-methylaminoanthraquinone, 1-ethylaminoanthraquinone, and 2-dimethylaminoanthraquinone. Alkylarylamines. According to the Provisional Specification, alkylamines of the benzene, naphthalene, and anthracene series are obtained by treating the amines with alkyl sulfates in an inert medium or solvent.

## II—BIOLOGICAL CHEMISTRY

HATTIE L. HEFT, EDGAR G. MILLER, JR., AND WILLIAM J. GIES

### A—GENERAL

FRANK P. UNDERHILL

**Chemistry's contribution to the life sciences.** A. S. LOEVENHART. *J. Ind. Eng. Chem.* 12, 1122-4(1920).—A review of the work of Lavoisier on combustion and oxidation and of Ehrlich on *saltarsan*. A chemical training cannot often be superimposed upon biological training but must precede it.

H. V. ATKINSON.

**The present status of the question of the internal secretions.** L. ASHER. Univ. Bern. *Deut. med. Wochschr.* 46, 1028-30, 1056-7(1920).—General considerations.

S. AMBERG

**Studies on the compensatory hypertrophy of the thyroid gland.** V. The effect of the administration of thyroid, thymic gland and tethalin and of a meat diet on the hypertrophy of the thyroid gland in guinea pigs. LEO LOEB. St. Louis. *J. Med. Res.* 42, 77-89(1920); cf. *C. A.* 14, 3456.—Feeding with thyroid tablets has a marked inhibiting effect upon compensatory hypertrophy of the thyroid gland. This effect is direct and sp. I preps. given simultaneously with thyroid tablets do not counteract the effect of thyroid. Feeding with thymus gland does not prevent thyroid hypertrophy. Repeated injections of tethalin do not noticeably influence the degree of thyroid hypertrophy which follows extirpation of the greater part of the gland. A diet consisting principally of meat given to guinea pigs during a period of three and one-half weeks does not produce hypertrophic changes in the thyroid gland, nor does it appreciably influence compensatory hypertrophy of the thyroid gland in the guinea pig.

E. B. FINK

**The significance of the gastric hydrochloric acid and remarks on the significance of the hydrogen-ion concentration in biological science.** I. TRAUBE. *Biochem. Z.* 107, 295-9(1920).—A brief critical review.

F. S. HAMMETT

The hydrogen-ion concentration of some standard solutions at different temperatures. L. W. WALBAUM. *Biochem. Z.* 107, 219-28(1920).—Using various combinations of Sørensen's "buffer" salt solns. and the colorimetric method of estn. of the H-ion concn., W. observed that with rising temp. the H-ion concn. of glycine-NaOH, borate-HCl and borate-NaOH also increased, while the citrate-NaOH mixts. underwent the reverse change. In general when the  $p_H$  falls with rising temp. the change is the most marked in the most alk. solns. Tables are given of the results. F. S. H.

Chemistry of the polysaccharides. E. HERZFELD AND R. KLINGER. *Biochem. Z.* 107, 268-94(1920).—Methods are briefly given for the prepn. in pure form of the higher polysaccharides starch, cellulose, agar, glycogen and inulin, and the soly. decompn.,  $I_2$ -reaction, ozazone-reduction and Ba(OH)<sub>2</sub> and tannin reaction are tabulated. The  $I_2$  reaction depends on the absorption of  $I_2$  on the surface of the colloid particles and the changes in color are related to the degree of dispersion of the latter: blue signifying a relatively coarse dispersion, red-brown a highly dispersed condition. Starch can be dextrinized by simple adsorption of its surface by solns. (e. g. formaldehyde) going from the gross to the finely state of dispersion. *Dextrins are not split products of starch, but are simply more highly dispersed starches.* The action of the diastatic enzymes is also considered to be a simple alteration of the degree of dispersion of the starch conditioned by the going into soln. mixts. whereby the previously insol. particles receive H<sub>2</sub>O-combining surfaces and become colloiddally divided. From this the grossly dispersed starch first goes over into smaller particles, ( $I_2$  positive dextrins) and these again into a still more disperse  $I_2$ -negative state *without hydrolysis being necessary.* No splitting to sugar could be observed as the consequence of the action of this enzyme. The enormous increase of surface is obviously an important prepn. for the later hydrolysis. The active materials of the diastase are apparently split products of the lipoids or protein bodies or their derivatives. The many polysaccharides of the plant world are not significantly different one from the other in either chem. or phys. properties: their apparent differences lying in the dextrin particles produced by the soln. mixts. and the presence or absence of foreign materials, such as protein, lignin, etc. Since the starch that has gone over to dextrin is sol. and can pass through membranes, the old conception of a starch-splitting into sugar and a rebuilding into polysaccharide is unnecessary. Animal glycogen is identical with the dextrinized starch, and its presence in the animal body is considered as due to the nature of the soln. mixts. preventing the survival of gross starch particles but little dispersed. F. S. HAMMETT

Oxidizing enzymes. II. The nature of the enzymes associated with certain direct oxidizing systems in plants. M. W. ONSLOW. Cambridge. *Biochem. J.* 14, 535-40(1920); cf. *C. A.* 13, 2886.—Three components are present in oxidase systems: A "catechol" compd. which may give rise to a peroxide, and 2 enzymes—an oxygenase which helps the formation of the peroxide, and the peroxidase, which decomposes the peroxide forming "active" O. The peroxidase may be partially sepd. from the oxygenase by fractional pptn. with alc. III. The oxidizing enzymes of some common fruits. *Ibid* 541-547.—The oxidizing enzymes present in the apple, quince, pear, plum, banana, orange, lemon, lime and raspberry were investigated. The author shows how divergent results may be obtained depending upon whether the test is performed on the fresh tissue, or on exts. prepd. in different ways. Such substances as org. acids, tannins, etc., may interfere with the reactions. BENJAMIN HARROW

Comparative determinations of the number of blood corpuscles, serum protein and sodium chloride in venous and capillary blood. BÖGENDORFER AND NONNENBRUCH. *Deut. Arch. Klin. Med.* 133, 389-96(1920).—It is shown with the usual methods that the capillary blood from the finger tip and the blood from the arm vein show wide differences in their content in erythrocytes, serum protein and NaCl. J. C. L.

Further study of the process of purifying pancreatic amylase. H. C. SHERMAN, I. D. GARARD AND V. K. LAMER. *J. Am. Chem. Soc.* 42, 1900-7(1920).—It has been shown (*C. A.* 14, 188) that attempts to purify pancreatic amylase by fractionation of pancreatin result in greatly concg. both the amylolytic and proteolytic activities in certain fractions but that there are also large losses of both types of activity. To det. whether, *e. g.*, the loss of amylolytic activity occurring when the partially purified amylase is dissolved and repptd. is due to incompleteness of pptn., to deterioration or, conceivably, to a change of amylase into protease, the process of purification was carried out with detns. of total solids and enzyme activity at each step wherever feasible. It was found that alc. up to 50% or EtOH-Et<sub>2</sub>O up to 8% by vol. of the substrate does not materially affect the activity of pancreatic amylase. When 50% alc. exts. are pptd. by EtOH-Et<sub>2</sub>O as in the usual purification process the residual soln. (Mother Liquor 1) contains about 1% of the active amylase and 2.5% of the solids of the original pancreatin, or about 5% of the solids originally extd. If the EtOH-Et<sub>2</sub>O ppt. is dispersed in H<sub>2</sub>O and mixed with abs. alc., 25% of the solids originally extd. remains in the filtrate (Mother Liquor 2) but this filtrate has no amylolytic activity. From 10-20% of the amylolytic activity accounted for is found in the dialyzates. In the purification expts. described about 60% of the amylolytic activity was lost by the end of the dialysis, about 45% in the final pptn. and sepn. and about 5% was found in the final ppt. By the use of liquid air cooling it has been found possible in other expts. to diminish the losses in the later stages and considerably to increase the amt. of active amylase recovered in the final product. The ppt. formed in the inner soln. during dialysis (Ppt. 3 or "sac ppt.") has very high proteolytic and very little or no amylolytic activity; this material, which was originally extd. by 50% alc., now ppts. from this solvent, probably owing to the removal, during the intervening purification process, of some substance or substances interfering with the coagulation of the protease or stabilizing its dispersion.

CHAS. A. ROUILLER

Sorption by cellulose (filter paper) and starch. Study of imbibition. K. SCHERINGA. Utrecht. *Pharm. Weekblad* 57, 1289-94(1920).—Absorption or adsorption by cellulose is largely due to its capillary structure. With many neutral salts there is practically no adsorption; heavy metals and alkaloids may be adsorbed from very dil. neutral soln. but not from acid soln.; alkalies may be adsorbed, partly due to impurities in the cellulose. Positive colloids (as metal oxide sols) are strongly adsorbed by coagulation, while negative colloids (as sulfide sols) are not, and so pass readily through filter paper. Gases are not perceptibly adsorbed. Albumin is adsorbed from very dil. acid soln. but not from urine, which probably contains substances which inhibit the adsorption. Filter paper may therefore be safely used to filter urine for albumin analysis. The above expts. were made with filter paper dried at 120°. Air-dried paper, which may contain as much as 20% moisture, gives different results (Evans, *J. Phys. Chem.* 10, 290(1906)). Starch, a much more disperse system than cellulose, shows selective adsorption; negative with NaCl, positive with CuSO<sub>4</sub> in very dil. soln. but negative in more concd. soln.

JULIAN F. SMITH

The uniformities of nature (FAWSITT) 2.

## B—METHODS AND APPARATUS

STANLEY R. BENEDICT

Sugar in the blood; A micro-chemical method of estimation. R. L. MACKENZIE WALLACE AND C. D. GALLAGHER. *Lancet* 1920, II, 784-5.—A micro-method for the estn. of sugar in the blood based upon the method of Folin and Wu. Blood may be obtained from the end of the finger and is absorbed on blotting paper weighed on a torsion balance before and after. The blood is dissolved in a measured quantity of H<sub>2</sub>O, the proteins removed by the method of Folin and Wu and the sugar estn. carried

out in a colorimeter against standard solns. A formula is given for the estn. of the % of blood sugar from the colorimetric readings.

E. B. FINK

**A study of some biochemical tests. II. The Adamkiewicz protein reaction. The mechanism of the Hopkins-Cole test for tryptophan. A new color test for glyoxylic acid.** W. R. FRARON. Cambridge. *Biochem. J.* 14, 543-54 (1920).—The controversy between Hopkins and Cole on the one hand and Rosenheim (*Biochem. J.* 1, 233 (1906)) on the other, as to whether the Adamkiewicz test for proteins really depends upon the presence of glyoxylic acid (as Hopkins and Cole claimed), and not upon formaldehyde (the view taken by Rosenheim), is decided in favor of Hopkins and Cole. "The Hopkins-Cole test may be carried out at temps. below that of the decompn. of the glyoxylate and in the absence of water, by using solid tryptophan and Ca glyoxylate. The characteristic color develops under conditions where formaldehyde can be shown to be absent. The new color test for glyoxylic acid is carried out as follows: The reagent used is 1% pyrogallol in pure concd.  $H_2SO_4$  (free from  $HNO_3$ ); it cannot be kept for more than a few days. A trace of Ca glyoxylate, or 1 or 2 drops of reduced oxalic acid give a deep blue color with the reagent when the mixt. is warmed. The addition of water changes the soln. to carmine; the blue color is again obtained by the addition of more  $H_2SO_4$ .

BENJAMIN HARROW

**New quantitative method for the determination of bromine.** G. HARTWICH. *Biochem. Z.* 107, 202-6 (1920).—100 cc. of urine are ashed with  $Na_2CO_3$  in a Ni crucible, the ash is dissolved in hot  $H_2O$  and the soln. filtered and made to about 50 cc. with washings. A 5 cc. portion of the filtrate is put in a shaking flask and  $H_2SO_4$  added until  $CO_2$  evolution stops. 20 cc. of Cl-water are measured from a dark buret into a small flask containing some KI soln. (without shaking); the buret is again filled and 2 cc. of the Cl-water measured into the soln. in the shaking flask which is immediately stoppered and shaken. Then 2 to 3 cc.  $CHCl_3$  are added, the whole is shaken and the  $CHCl_3$  is let out onto a watch glass. If the  $CHCl_3$  contains free Br it takes on a yellow tint and becomes violet colored on the addition of 3 to 5 drops of an indicator soln. made of 10 cc. of a 2% alc. fuchsin plus 10 cc. 0.1 N  $H_2SO_4$  and 80 cc.  $H_2O$ ; if no Br is present the above reaction does not occur, and the process is repeated, using separate watch glasses for each extn. Violet coloration usually occurs on the 2nd extn. and extns. are continued until no reaction is given. The end-point is snappy. The number of watch glasses shows how many 2 cc. lots of  $CHCl_3$  were used to obtain all the free Br. The control is titrated with 0.1 N  $Na_2S_2O_3$  soln., thus giving the titer of the Cl-water, and the Br content of the urine is detd. according to the formula,  $Br = (Ur)(Cl)(T)(F)/(ur)$  (12.5), in which  $Ur$  = total urine vol.;  $ur$  = vol. of urine ashed,  $T$  = amt. of 0.1 N  $Na_2S_2O_3$  soln. used for 20 cc. Cl-water,  $F$  = vol. of filtrate from ashing,  $Cl$  = twice the no. of watch glasses and 12.5 = factor when 5 cc. filtrate and 2 cc.  $CHCl_3$  are used. Each detn. takes from 10-20 min. and the errors of manipulation are very slight. Controls run remarkably close; some of the figures are: Present 75.3 mg., found 75.5 mg.; present 345.4 mg., found 345.4 mg.

F. S. HAMMETT

**The influence of the color of the urine on readings of the phenolsulfonephthalein test.** V. O. PEDERSEN. *N. Y. Med. J.* 112, 477-83 (1920).—An extensive tabular presentation of data obtained in an attempt to account for the apparent loss of 15% of the dye as indicated on the scale between the 85% of the material excreted by the patient in 2 hrs. and the 100% of the phenolsulfonephthalein that has been injected, from which it is concluded that the color of the urine interferes markedly with the accurate detn. of the excreted dye. Using Vogel's scales as indices for comparison, it was found that in urines equiv. to Scale 4 the av. error is at least 15% and probably 20% with the darker urines, while with Scale 3 the error is at least 10% and may run as high as 15%. With Scales 2 and 1 the error is about 10%. P. therefore, considers

that in all the ordinary scale readings 10% should be deducted for pale yellow, 15% for positive yellow and 20% for urines with a reddish or orange tinge. F. S. H.

**The urea output as a practical kidney function test.** S. H. BLODGETT. *N. Y. Med. J.* 112, 483-6(1920).—A discussion and report of exptl. studies showing that the ability of the kidney to pass off the waste products of metabolism is not, in many cases, shown by any of the so-called kidney function tests, while this is easily discovered by means of feeding definite amounts of nitrogenous food to a person previously put on a so-called basic diet and watching the output of urea. Having this knowledge the diet can then be built up so that the person takes the max. amount of nitrogenous food, the waste from which his kidneys can get rid of. F. S. HAMMERTT

**A simple apparatus for obtaining blood specimens.** JOHN W. HART. *Public Health Reports* 35, 459-60(1920).—An ordinary test-tube is drawn out until it has an opening of  $\frac{1}{4}$  in. It is then connected to a small glass or metal stopcock by means of rubber tubing. The other end of the stopcock is connected to a 15 cc. all-metal syringe. When a vacuum is secured in the test-tube by pulling out the syringe plunger the stopcock is closed, and the syringe disconnected and in its place a luer needle is connected. When the needle is inserted in the vein the stopcock is opened and the blood is drawn into the tube by the vacuum. JULIAN H. LEWIS

**Modifications of Van Slyke's titration method for estimating the alkali reserve of blood.** HOWARD D. HASKINS AND EDWIN E. OSGOOD. *Univ. Oregon. J. Lab. Clin. Med.* 6, 37-41(1920); *C. A.* 13, 2052.—The clear standard of the Van Slyke method is replaced by a turbid one containing starch. The technic of the operation is as follows: 5 cc. of blood is drawn into a paraffined centrifuge tube containing 0.07 cc. 30% (COOK)<sub>2</sub> soln. and 1 cc. of paraffin oil. Two cc. of the centrifuged plasma is rotated in a flask with 5 cc. 0.02 N HCl and a drop of caprylic alcohol. The mixt. is washed into a 120 cc. Florence flask with 20 cc. distilled water. 3 cc. neutral red soln. is added and the mixt. titrated to the proper end-point with CO<sub>2</sub>-free 0.02 N NaOH, comparing the end-point with a turbid standard. The latter is prepared as follows: To 60 cc. of buffer phosphate soln. ( $P_H$  6.8) add 5.6 cc. of amaranth soln. (dissolve 8 mg. in 100 cc. distd. water and add 0.5 cc. chloroform-thymol and 5.2 cc. of paranitrophenol soln. (dissolve 20 mg. in 10 cc. alcohol and dilute to 100 cc. with distilled water). Transfer 30 cc. of this mixt. to a 120 cc. nonsol Erlenmeyer flask, and add 20 mg. of dry, finely powdered cornstarch and 0.2 cc. of chloroformthymol preservative. To calculate the alkali reserve the titration value of 5 cc. of the N/50 HCl minus the number of cc. of 0.02 N NaOH used is multiplied by 22.4. Values closely approximating the figures obtained with the Van Slyke apparatus for detg. the CO<sub>2</sub> combining power of the blood were obtained in 15 cases. The agreement where a clear standard was used was not so close. E. R. LONG

**Improved test for the detection of glucose, especially in urine.** W. S. HAINES, G. P. POND AND R. W. WEBSTER. *J. Am. Med. Assoc.* 74, 301-2(1920).—The improved Haines soln. contained CuSO<sub>4</sub> 5 g., glycerol 250 cc., KOH 20 g. (or NaOH 14.3 g.), water to 1000 cc. The CuSO<sub>4</sub> is dissolved in the glycerol with the addition of an equal vol. of water, the KOH in about 200 cc. of water is added to the Cu soln. with stirring, and the mixt. made up to 1000 with water. Some samples of glycerol cause reduction; in such cases let the soln. stand 48 hrs. then decant or filter the clear from the Cu<sub>2</sub>O ppt. A test is made as follows: Heat about 5 cc. of the Cu soln. to boiling, remove from flame, incline the tube and float on 10 to 20 drops of urine freed from phosphates by KOH or NaOH. The tube is then righted and the reaction noted. If sugar is present in amts. exceeding 0.1% a brick red or yellowish ring appears at the junction of the liquids. Smaller amts. down to 0.03% will show a ring within one min. and a more yellow color. Urines containing no pathologic sugar give no ring.

The reaction is concd. to a single plane, thus increasing its visibility. Results are prompt and but one heating is necessary. L. W. RIGGS

**Indican in serum.** I. SNAPPER AND VAN VLOTEN. *Nederlandsch Tijdschrift v. Geneeskunde*, July 10, 1920, 136; *J. Am. Med. Assoc.* 75, 1034.—This paper emphasizes the importance of the normal indican level in the blood serum, as an excess points to deficient functioning of the kidneys. Any excess is readily detd. by treating 3 or 4 cc. of serum with equal parts of a 20% soln. of  $\text{CCl}_3\text{COOH}$  to remove albumin; 2. 5 cc. of the filtrate is dild. to 10 cc. with water. Then 1 cc. of thymol spirit and 10 cc. of the Obermeyer reagent are added. After 20 min. the tube is shaken with 2 cc.  $\text{CHCl}_3$  and in 30 min. the  $\text{CHCl}_3$  turns pink if the indicanemia is above normal.

L. W. RIGGS

**Potato-spirit vs. carbide spirit.** HENNIG. *Z. Spiritusind.* 43, 299(1920).—Polemical against J. Hess, who upheld in an article in the "*Münchener Neuesten Nachrichten*," the practical possibility of producing alc. from carbide. W. B. V.

**A new technical method for the estimation of the saccharogenic power of diastatic preparations.** KOKICHI OSHIMA. *J. Ind. Eng. Chem.* 12, 991-3(1920).—If the sample to be tested is a solid 1 to 10 g. are extd. with 100 cc. distd.  $\text{H}_2\text{O}$ , 10 cc. of this ext. put into a flask containing 100 cc. of sol. starch soln. and held at  $40^\circ$  for 30 min. Then 10 cc. of 0.2 N NaOH are added to stop enzyme action and graduated quantities of this mixt. added to a series of test-tubes containing 0.5 cc. of Fehling's soln. The smallest amt. of the digested starch soln. which just reduces 5 cc. of Fehling soln. is detd. and from this the saccharogenic power of the prepn. calcd. usually in terms of the Lintner scale. The values obtained by this method for a culture of *Aspergillus oryzae* and for an ext. of malt are calcd. to the Lintner scale and given in the form of tables. The Lintner value is proportional to the time of digestion. This method is convenient for such exptl. studies as the influence of alkalinity, acidity, etc., on the activity of diastase prepn's. G. W. STRATTON

The salt error of cresol red (WELLS) 7.

AGASSE-LAFONT, E.: *Les applications pratiques du laboratoire à la chimique. Principes, techniques, interprétation des résultats.* 3rd Ed. Paris: Vigot frères. 992 pp F. 50. For review see *Bull. sci. pharmacol.* 27, 553(1920).

## C—BACTERIOLOGY

A. K. BALLS

**Application of the hydrogen-ion theory to the determination and adjustment of the reaction of culture media.** A. BESSEMANS. *J. Pharm. Belg.* 2, 833-40(1920).—A review of the colorimetric methods which have been proposed for the detn. of the H-ion concn. with special reference to the detn. of the H-ion concn. of culture media, and of the methods of adjusting media to a desired H-ion concn. A. G. DUMÉZ

**The possible pathogenicity of *Bacillus botulinus*.** RUTH B. EDMONDSON, L. T. GILTYNER AND CHARLES THOM. U. S. Dept. Agr. *Arch. Internal Med.* 26, 357-86 (1920).—Following the technic of Bullock and Cramer (*C. A.* 13, 1727) with *B. welchii* and with *B. tetani*, E., G. and T. found that  $\text{CaCl}_2$  acts as an accessory factor in producing botulism when injected into guinea pigs with toxin-free bacilli or spores of *B. botulinus* (Boise strain). The pathogenicity of the Nevin strain was not increased in this manner. When the washed and heated spores were fed to guinea pigs in sufficiently large doses it was found that the Boise strain produced the characteristic symptoms and death and the Nevin strain occasionally did so. "Food suspected of containing *B. botulinus* should be destroyed, not heated and eaten." I. GREENWALD

**The production of acetaldehyde by certain pentose-fermenting bacteria.** W. H. PETERSON AND E. B. FRED. Univ. Wis. *J. Biol. Chem.* 44, 29-45(1920).—In the presence of  $\text{Na}_2\text{SO}_3$  or  $\text{CaSO}_3$ ,  $\text{MeCHO}$  is formed in the fermentation of glucose, starch



and xylose by *Lactobacillus pentoceticus*, *Bacillus acetoethylicum* and an organism related to the colon-aerogenes group and known as Culture 28, the yield being greatest with xylose. In solns. containing 2% of the carbohydrate, the yield varied from 0.020 to 0.157 g. per 100 cc. and was proportional to the amt. of fixative used.  $\text{Na}_2\text{SO}_4$  was more effective than  $\text{CaSO}_4$  but the amt. that could be used was limited because of its alkalinity. An increase in the amt. of  $\text{MeCHO}$  formed was accompanied by a decrease in the amt. of  $\text{EtOH}$  and an increase in the amt. of volatile acid.  $\text{MeCOCO}_2\text{H}$  was fermented to some extent by all 3 organisms, least by *Lactobacillus pentoceticus* and most by Culture 28 with the formation, with or without the presence of  $\text{Na}_2\text{SO}_4$ , of volatile acid,  $\text{CO}_2$  and  $\text{H}_2$ , but no  $\text{MeCHO}$  could be detected. I. GREENWALD

The action of surface-active nonyllic acid and some surface-active higher homologs of the alcohol series (amyl alcohol and octyl alcohol) on the yeast cell and fermentation. W. WINDISCH, W. HENNEBERG AND W. DIETRICH. *Biochem. Z.* 107, 172-90(1920).—Continuing the previously reported studies the expts. here detailed show that nonyllic acid, acting in its capacity as a surface-active substance, in increasing amts. from 0.005 to 0.02% at first acts as an accelerator and then as an inhibitor of fermentation. The latter effect is evident by the death of the yeast cells accompanied by changes in the shape of the cells and fat formation. Similar effects are obtained when octylalc. in concns. from 0.017 to 0.04% are used, smaller amts. than 0.017% producing the changes in structure previously noted. The action of these compds. is said to be due to their surface activity and not to their chem. properties. F. S. HAMMETT

The antiseptic action of some chlorine derivatives of methane, ethane and ethylene. E. SALKOWSKI. *Biochem. Z.* 107, 191-201(1920).—These briefly reported expts. show that  $\text{CH}_3\text{Cl}$ ,  $\text{CHCl}_3$ ,  $\text{CH}_2\text{Cl}_2$ ,  $(\text{CH}_2\text{Cl})_2$ ,  $\text{CHCl}-\text{CCl}_2$  and  $\text{C}_2\text{H}_4\text{Cl}_2$ , all possess antiseptic properties, some to a greater degree than others when used as preservatives of biological products. F. S. HAMMETT

The pharmacology of selenium and tellurium. I. The action of their acids on bacteria. GEORGE JOACHIMOGLU. *Biochem. Z.* 107, 300-12(1920).—Report of studies of the action of various concns. of selenic and selenous, and telluric and tellurous acids on widely divergent types of bacteria, which demonstrates that corresponding to the relations found between arsenic and arsenious acids the ions of selenous and tellurous acids are much more effective than are the ions of the selenic and telluric acids. While *Hyphomycetes* are scarcely at all influenced as to growth by either tellurites or tellurates, bacteria are relatively susceptible. The most susceptible are the bacteria of the typhus-coli group which are strongly inhibited in growth of concns. of 1:800,000. F. S. HAMMETT

A calcium and iron metabolizing bacillus. A. BRUSSOFF. *Centr. Bakt. Parasitenk., Abt. II* 48, 193-210(1918).—A bacilliform bacterium is described which will take up Fe and Ca salts from the media. JULIAN H. LEWIS

The preparation of culture media with "extract of Magermilch." W. PFÄRLER. *Centr. Bakt. Parasitenk., Abt. I* 83, 298-9(1919).—"Extract of Magermilch" a patented product is a fairly satisfactory substitute for meat exts. JULIAN H. LEWIS

The Kindborg acid-fuchsin agar. GUSTAV GASZNER. *Centr. Bakt. Parasitenk., Abt. I* 83, 301-4(1919).—The decolorization of acid-fuchsin agar by typhoid and similar organisms is not due to reduction of acid fuchsin, as thought by Kindborg, but is due to the production of alkali from the proteins of the culture media. J. H. L.

Coagglutination in cholera. STANISLAW SIERAKOWSKI. *Centr. Bakt. Parasitenk., Abt. I* 84, 178-90(1920).—The cholera vibron can be differentiated from similar organisms by the absorption of coagglutinins by the latter organisms which can be sepd. into 4 groups. The nonsp. organisms will absorb the agglutinins for themselves without affecting the agglutinins for the cholera organism. JULIAN H. LEWIS

The relation of the agglutinins for *Bacillus proteus* X-19 and the Typhoid-dysentery bacilli. FELIX E. R. LOEWENHARDT. *Centr. Bakt. Parasitenk., Abt. I* 84, 191-201 (1920).—In typhus fever the Co agglutination phenomenon, which consists of an agglutination of the non-pathogenic organism *B. proteus* X-19, is explained by the presence of common receptors in the organism of typhus and the proteus bacillus. However, the coagglutination of the typhoid bacillus in typhus is the result of a nonsp. stimulation of the antibody producing centers already sensitized to typhoid. J. H. L.

The disinfectant properties of various wall paints. FRITZ CRONER. *Centr. Bakt. Parasitenk., Abt. I* 84, 314-20(1920).—The powers of various wall paint, colors to kill bacteria are compared. The lime-prepns. were least effective, the glue-prepns. and more so the best prepns. were patented mineral tints. JULIAN H. LEWIS

The action of several chemical disinfectants on tetanus spores. THEOD. MOLL. *Centr. Bakt. Parasitenk., Abt. I* 84, 416-34(1920).—The disinfectant value of a chemical against tetanus spores cannot be stated unless something is known about the heat resistance of the spores. This heat resistance is very changeable and dependent on the kind of the culture, the compn. of the media, and the age of the spores. To make the adherent toxin of the spores unharmed they must be heated to 65° for 5 mins. Strongly oxidizing substances, as the halogens and  $H_2O_2$ , have a marked disinfectant action on tetanus spores, while the protein pptd. substances, as alc., heavy metals, phenol and cresol, have slight or no disinfectant action. JULIAN H. LEWIS

Several new disinfectants. II. FRITZ DITTHORN. *Centr. Bakt. Parasitenk., Abt. I* 84, 486-96(1920).—A no. of cresol prepns. offered as substitutes for cresol-soap prepns. are described and their properties investigated. JULIAN H. LEWIS

Comparison of the methods of Herman and of Ziehl-Neelson for staining tubercle bacilli. ELIZABETH KONGSTED. *Centr. Bakt. Parasitenk., Abt. I* 84, 513-5(1920).—The Herman method is as follows: The stain consists of 1 part 3% crystal-violet soln. in 95% alc. and 3 parts 1%  $(NH_4)_2CO_3$  in water. One drops 6-8 drops of the stain on the prepn. and heats 1 min. to boiling. Decolorize several secs. in 10%  $NaNO_3$  and then 95% alc. until a pale blue. Counterstain in 1/4% eosin in alc. soln. or a weak aq. soln. of picric acid. K. examd. 1200 smears, of these 345 were positive. And of this no. 29 were positive only with the Herman stain and 8 only with the Ziehl-Neelson stain. JULIAN H. LEWIS

The acid precipitation of *Proteus* strains. F. W. BACH. *Centr. Bakt. Parasitenk., Abt. I* 84, 265-79(1920).—The acid pptn. method of Michaelis is not adapted to the differentiation of typhus-specific and nonsp. strains of the *Proteus* group. The H forms of the X strain show a great agreement in physico-chem. properties, especially in acid pptn. The H forms of the nonsp. strains show on the other hand a great individual variation, probably because of differences in the inner structures of the cells. The O forms of all strains, those from carbol-agar, as well as spontaneous growing forms, show irregular changing relations to the minimal acid pptn. concn. The pptn. results differentiate them from the H form. The serum agglutination and acid agglutination go parallel, but they need not be identical. Acid pptn. gives no conclusion *a posteriori* to serum agglutinability and *vice-versa*. Acid agglutination may show similarity or differences of various strains of the same species of bacteria in their relation to physico-chem. influences, without showing an analogous relation to serum agglutination. JULIAN H. LEWIS

Observations on changes in virulence of hemolytic streptococci with special reference to immune reactions. Y. NAKAYAMA. *J. Infect. Dis.* 27, 270(1920).—The agglutinability of a streptococcus may change as the result of animal passage, the particular strain used for immunization being agglutinated more strongly than the related strains by the corresponding immune serum. The original nonvirulent mother streptococcus was agglutinated by all the immune serums. The same relation seems to ob-

tain with reference to opsonins and phagocytosis, as well as with respect to sp. pptn., and conglutination, but no differences could be made out between the different strains by means of complement fixation. All the various strains were agglutinated in the same way by acid soln.

JULIAN H. LEWIS

The relation between the number of bacteria and acid production in the fermentation of xylose. J. A. ANDERSON, E. B. FRED AND W. H. PETERSON. *J. Infect. Dis.* 27, 281-82(1920).—The rate of acid production in the fermentation of xylose by *Lactobacillus pentoceticus* is most rapid during the period of max. growth of the bacteria. A decline in the rate of growth is accompanied by a decrease in acid production although a slow acid formation is noted for many days. In the first stages of growth, the curves of multiplication of bacteria and of acid formation are almost parallel although growth precedes the formation of acid in measurable quantities. The max. no. of bacteria occurs in the early stages of fermentation, usually within 48 hrs. after inoculation, while the max. acidity is not noted for several days. In a xylose yeast-water medium the bacteria multiply more rapidly and reach a higher no. if  $\text{CaCO}_3$  is present. In old xylose cultures of *Lactobacillus pentoceticus* the ratio between lactic and acetic acids may change. This is probably due to a secondary fermentation of the lactic to acetic acid. Apparently, acetic acid is the only volatile acid produced in the fermentation of xylose by *Lactobacillus pentoceticus*. Direct count and plate count of the total no. of bacteria give the same general results.

JULIAN H. LEWIS

Further observations on varieties of streptococci with reference to hemolysis. B. J. CLAWSON. *J. Infect. Dis.* 27, 368-77(1920).—All of the 50 hemolyzers tested produced a green discoloration of the sheep red cells on heated blood similar in all respects to the green produced by nonhemolyzers. This green colored substance seems to be methemoglobin when compared with the green produced on the blood-agar plate by *Streptococcus viridans* and according to the spectroscopic test. Methemoglobin is more readily produced by hemolytic strains of streptococci in heated blood than in nonheated blood.

JULIAN H. LEWIS

A new cresol compound. F. NEUFELD AND O. SCHJEMANN. *Z. Hyg.* 85, 193-4. Because of the scarcity of soap, several cresol preps. containing no soap have been manufd. to take the place of lysol. Among them are preps. known as phenolul, cresotincresol and betalysol. They prove to be much inferior to lysol and conditions for their use are given.

JULIAN H. LEWIS

Bacterial vaccines—chloretone solution as a vehicle for their administration. R. G. OWEN, F. A. MARTIN AND W. L. BROSIUS. Detroit. *J. Lab. Clin. Med.* 6, 47(1920).—Bacterial suspensions are made by washing organisms from agar slants with satd. chloretone soln., diluting to the required degree, and heating to 55-60° for 1 hr. There is no clumping of organisms as with tricresol preparations. The anesthetic properties of chloretone make the use of such a vaccine most acceptable to the patient. The chloretone preserves sterility.

E. R. LONG

A starch-splitting enzyme bacterium found in cases of diabetes mellitus. W. FORD ROBERTSON. *J. Path. Bact.* 23, 122-3(1920).—An anaerobic streptothrix was found in great abundance in the stools of 8 consecutive cases of diabetes; it had the power of splitting starch into substances which will reduce Fehling soln. Preliminary feeding expts. indicate that an intermittent glucosuria may be produced in rats and rabbits.

JOHN T. MYERS

A method for the simultaneous demonstration of Gram-positive and Gram-negative organisms in sections. G. HASWELL WILSON. *J. Path. Bact.* 23, 123-4(1920).

JOHN T. MYERS

The antiseptic properties of compounds of the acridine and phenazine groups. C. H. BROWNING, J. B. COHEN AND R. GULBRANSEN. *J. Path. Bact.* 23, 124-5(1920).—The introduction of amino groups into acridine and dimethyl-acridine increases its

antiseptic potency for staphylococci and *B. coli*. The methyl chloride is more generally potent than the hydrochloride in serum. Effectiveness in serum is a characteristic of the amino compds., especially in the case of the methyl chlorides. The lethal concn. for staphylococcus reached 1:100000 only in the case dimethylamino-amino-methyl-phenazine (toluylene-red), and for *B. coli* did not exceed 1:15000 for any of the compds.

JOHN T. MYERS

Influence of the nature of carbonaceous food on the utilization of nitrogen by *Bacillus subtilis*. E. AUBEL. *Compt. rend.* 171, 478-80(192).—*B. subtilis* is readily grown on a medium containing asparagine 6 g., potassium phosphate 1, MnSO<sub>4</sub> 1, and water up to 1000, in which asparagine is the sole source of both C and N. If in such a medium the source of C is varied a marked difference results in the amt. of N utilized and in the wt. of the growth. Addition of glycerol to the medium increased the proportion of N utilized more than the addition of levulose, and the latter more than glucose. This favorable action of glycerol was attributed to the formation of pyruvic acid and this idea was supported by results obtained by adding sodium pyruvate to a control culture. It appears that C from compds. containing the ketone group has the most favorable action, then aldehydic and finally C attached directly to H. L. W. RIGGS

#### D—BOTANY

CARL L. ALSBERG

Inulin-producing plants. H. COLIN. *J. Fabr. Sucre* 61, No. 16(1920).—The inulin content of dahlia tubers, Jerusalem artichoke tubers, and chicory roots at different times of the year is given. In all cases it is highest in early autumn. L. E. G.

Composition of gases in intercellular spaces of apples and potatoes. J. R. MAGNESS. *Bot. Gaz.* 70, 308-16(1920).—M. describes an app. which enables one to obtain a sample of the gas within the tissues, without contamination with air. Using apples, the amt. of CO<sub>2</sub> and O varies greatly with temp. At 2° the % of CO<sub>2</sub> is 6.7 and that of O 14.2; at 30° it is 21.4 and 3.2, resp. The total CO<sub>2</sub> and O is much higher in potatoes than in apples. Removing the epidermis facilitated the entrance of O to the tissues, and also the escape of accumulated CO<sub>2</sub>. Three factors operate to det. the amts. of CO<sub>2</sub> and O in the intercellular spaces at any given temp.: (1) Rate of oxidation; (2) permeability of the skin to CO<sub>2</sub> and O; (3) difference in pressure of CO<sub>2</sub> and O within and without the fruit (which det. the rate of gaseous exchange when the permeability factor is const.).

BENJAMIN HARROW

Cross-inoculation studies with the nodule bacteria of lima beans, navy beans, cowpeas and others of the cowpea group. A. L. WHITING AND ROY HANSEN. *Soil Science* 10, 291-300(1920).—Pot expts. with leguminous plants and pure cultures of *B. radicola* show that the nodule bacteria of the lima bean (*Phaseolus lunatus*) are distinct from those of the navy and kidney beans (*Phaseolus vulgaris*) but are identical with those of the cowpea group.

W. J. ROBBINS

A mosaic disease of cabbage as revealed by its nitrogen constituents. S. L. JODIDI, with S. C. MOULTON AND K. S. MARKLEY. *J. Am. Chem. Soc.* 42, 1883-92 (1920); cf. *C. A.* 14, 2012.—Under normal physiological conditions the % of total N in the leaves is very much higher (up to 107%) than in the roots, while in diseased leaves and roots the difference is essentially smaller (about 70%). While the difference in N content between normal and diseased leaves is considerable (27-33%), for the roots, the difference is comparatively small (7-9%). Nitrites are found in diseased material only. Healthy leaves have a higher % of nitric N than the roots, whereas the reverse is true for diseased plants. While, absolutely, the % of nitric N in diseased leaves and roots is smaller than in healthy tissues, the nitrate ratio of the diseased to healthy roots is relatively very much greater than that of diseased to healthy leaves. Normally the leaves are richer in protein and protein N than the roots when calcd. on the basis

of the oven-dried cabbage; healthy tissues, however, always have a lower protein and protein N content than diseased tissues. The proportion of acid amide, basic and non-basic N is higher in healthy than in diseased plants. No definite conclusion can be drawn as to the peptide N. The % of humin N appears to be higher in diseased than in normal plants. Judging from the striking similarity of the above analytical evidence with that obtained in the case of healthy and blighted spinach and taking into consideration that spinach blight belongs to the type of mosaic diseases it appears logical to relate the cabbage disease caused by denitrification to the latter type. C. A. R.

**Experiments on soaking seed.** M. P. BACHELIER. *Compt. rend. agr. France* 60, 712-16(1920).—Barley after soaking for 8 hours was planted in 3 series of plots. Treatment was as follows: (1) Grain not soaked; grain soaked in: (2) pure water; (3) in  $\text{KNO}_3$  (100 g. per l.); (4)  $\text{KNO}_3$  (200 g. per l.); (5)  $\text{NH}_4\text{NO}_3$  (100 g. per l.); (6)  $\text{NH}_4\text{NO}_3$  (200 g. per l.); (7) grain sprinkled with a soln. of  $\text{NH}_4\text{NO}_3$  (200 g. per l.).  $\text{NH}_4\text{NO}_3$  was shown to have a greater injurious action than  $\text{KNO}_3$ , while the best results were obtained by unsoaked seeds or seeds soaked in pure water. An experiment was conducted with spring wheat in which 20 g. of  $\text{KNO}_3$  per l. were used. In this case the action of the salt probably was not detrimental. F. M. SCHERTZ

**The effect of salts of boron upon the distribution of desert vegetation.** KARL F. KELLERMAN. *Bur. Plant Ind. J. Wash. Acad. Sci.* 10, 481-6(1920).—Address of the President of the Bot. Soc. of Washington, Feb., 1920. It is pointed out that the experience of the past 2 seasons in the use of fertilizers contaminated with borax, has drawn attention to the importance of considering B compounds not only in fertilizer investigations but also in investigations of alkali deposits wherever agr. developments are to be considered. A review is given of the recorded borax deposits of the U. S. K. found borax to occur in Kern Co., in the water of Lake Elsinore, Riverside Co., Calif., and the clear water lakes of the Southwest, associated with the common alkali salts. It is suggested that the lack of vegetative growth may be associated with the presence of B and that when lakes are free from vegetation, other conditions being suitable for growth, they should be examd. for the occurrence of borax. J. J. SKINNER

**Possibility of the formation of hexamethylenetetramine in assimilative plants and a microchemical reaction for ammonium salts.** C. VAN ZIJP. *Buitenzorg. Pharm. Weekblad* 57, 1345-8(1920).—The I-KI reagent (C. A. 12, 889) is applicable to salts of  $(\text{CH}_3)_2\text{N}_4$  as well as to the free base.  $\text{NH}_4$  salts do not interfere. To detect  $(\text{CH}_3)_2\text{N}_4$  salts in faintly acid soln., evap. over CaO and add a little I-KI soln. to the residue. Fine prisms of varying form and size (length up to about 0.1 mm.) are formed. The same crystals may be obtained by treating  $\text{NH}_4\text{Cl}$ ,  $(\text{NH}_4)_2\text{SO}_4$ ,  $(\text{NH}_4)_2\text{PO}_4$  or  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  in aq. soln. with formalin, evap. in air and adding I-KI soln. This shows that  $\text{CH}_3\text{O}$  reacts with  $\text{NH}_4$  salts to form  $(\text{CH}_3)_2\text{N}_4$  salts. It even reacts with the insol.  $\text{MgNH}_4\text{PO}_4$ , the products being  $\text{MgHPO}_4$  and  $(\text{CH}_3)_2\text{N}_4$  probably the free base. Since I-KI soln. is sensitive to 0.3 mg. of  $(\text{CH}_3)_2\text{N}_4$ , which requires less than half its wt. of  $\text{NH}_3$  for formation, the reaction affords a very sensitive test for  $\text{NH}_4$  salts.  $\text{CH}_3\text{O}$  reacts not only in alk. or neutral, but even in slightly acid soln. The conclusion is that  $(\text{CH}_3)_2\text{N}_4$  is probably formed during assimilation in plants. JULIAN F. SMITH

**The gum-disease of sugar cane, its cause and its control.** G. WILBRINK. *Arch. Suikerind.* 28, 1399-1525(1920).—The macroscopic and microscopic symptoms of the disease are described in detail. In the mycological investigation it was found that cane showing the symptoms of the disease always contains one and the same bacteria. Ordinary culture methods have so far given poor results with this organism, and pure cultures are best obtained by first inoculating a healthy plant, and then isolating the bacteria from the young vascular bundles of the latter. The organism differs in the following respects from that described by Groenewege (C. A. 10, 1363) as the causative agent of the Java gum-disease. It has a light yellow color in incident light, and is a

transparent brown in transmitted light; there is no capsule formation and no abundant production of slime;  $\text{NH}_4$ -salts, nitrates, and asparagine are not assimilated; milk is not coagulated, and there is no growth on its surface; the growth in 5% glucose soln. is slow, and in 8% soln. it stops entirely; the organism does not grow in 5% malt extract with 2% agar; it does secrete invertase, but not zymase. The thermal death point is  $52^\circ$ . It is undoubtedly different from the bacteria studied by Groenewege, but very similar to that described by R. Greig Smith and by Erwin F. Smith. The gum-disease can be produced in healthy cane by inoculation with pure cultures of the organism, and the bacteria can again be isolated from inoculated plants; this proves that it causes the disease. The latter is propagated principally, if not exclusively, through the medium of diseased cane. It is readily transferred to seed cane and stubble by cutting and topping with infected knives or by injuring cane with infected implements. Factors like soil, climate, fertilization, water, etc., have no causal connection with, but only an indirect effect on the disease, although there may be other ways of transmitting it than those mentioned. Only healthy cane must be used for planting, and the knives used should, after each cut, be dipped into a 5% lysol soln. It appears doubtful that the gum-disease of Java is identical with that found in Australia, Brazil, and Mauritius, because slime formation is only very slight. Importation of cane from the latter countries into Java should, therefore, be avoided. Neither is the gum-disease identical with serch or with the slime disease caused by *B. solanacearum*. Disinfecting the tools which come in contact with the plants affected by these diseases may also prove helpful in controlling them.

F. W. ZERBAN

Ability of plants to synthesize optical antipodes (Hass) 10.

#### E—NUTRITION

PHILIP B. HAWK

##### NORMAL

Calcium metabolism of infants and young children and the relation of calcium to fat excretion in the stools. II. Children taking a mixed diet. L. E. HOLZ, A. M. COURTNEY AND H. L. FALES. Rockefeller Inst. *Am. J. Diseases Children* 19, 201-22 (1920); cf. *C. A.* 14, 1361.—Ca excretion means here the total amt. of Ca in the stools, Ca absorption, the difference between the intake and this excretion. The summary states: With an adequate intake of Ca children (1-7 years of age, 79 cases) on a mixed diet absorb on an av. 0.055 g. CaO per kg., which is less than that of infants taking modifications of cow milk. The av. daily excretion was 0.87 g. of CaO. The av. intake was 0.108 g. per kg., which is lower than that of infants. With an intake of more than 0.09 g. per kg., the absorption nearly always exceeded 0.03 g. per kg., with an av. of 0.055 g. With a lesser intake the absorption rarely exceeded 0.03 g. per kg., the av. being only 0.015 g. with a negative balance in several cases. With an intake of CaO over 0.09 g. per kg. the absorption averaged about 40%; with an intake of 0.09 g. per kg. or less the absorption averaged about 20%. Children require an intake of at least 0.09 g. CaO per kg. The best absorption occurred when the intake of fat exceeded 3.0 g. per kg. and when, at the same time, for every g. of fat the diet contained 0.03-0.06 g. CaO. The addition of  $\text{CaCO}_3$  to the diet increased the absorption, that of Ca acetate or  $\text{CaHPO}_4$  did not. With a very small Ca intake the absorption did not exceed much the amt. normally excreted in the urine or there was a negative balance. The av. absorption of CaO per kg. was somewhat lower in larger children, but the age, irrespective of the wt., had no const. relation to the Ca absorption. The excretion of Ca in the stools was not related to the excretion of total fat, but has some relation to the excretion of fat as soap. In constipated stools, though, containing the most soap, on an equal intake of Ca, the Ca excretion was less than in the normal stools. The Ca lost as soap was, in most cases, an insignificant part of the Ca intake. The 2 factors

chiefly affecting the percentage of Ca in the stools of children were the amt. of Ca intake and the reaction of the stools. In children with *chronic intestinal indigestion* (21 cases about 2-8½ years old) the absorption of Ca was low. The absorption in cases of active rickets (5 cases) was lower, during recovery (10 cases) higher than normal. *Cod liver oil* increased the absorption of Ca except in cases where the intake of Ca or fat was very low. The substitution of vegetable fats for milk fat did not affect the Ca metabolism of children taking a mixed diet.

S. AMBERG

**The effect of high-protein and high-energy rations in feeding dairy cows.** W. B. ELLETT AND C. W. HOLDAWAY. Virginia Agr. Expt. Sta., *Tech. Bull.* 20, 3-16 (1920).—Two groups of animals were used, 1 received a ration of 9 lbs. of cornmeal, 2 lbs. of bran and 40 lbs. of silage; a high-energy ration, with a nutritive ratio of 1:11.0. The other group received a high-protein ration, consisting of 2 lbs. of cottonseed meal, 2 lbs. of bran, 7 lbs. of corn gluten meal and 40 lbs. of silage; the nutritive ratio being 1:2.4. Both rations supply the full necessary amts. of nutrients for maintenance and milk production; but the first contains an excess of energy, and the second, an excess of protein. In both groups at the time of the trials the requirements for digestible protein, net energy and dry matter were supplied by the rations when calcs. are made with av. digestion coefficients. During the expts. of the high-energy cows there followed a diminution of the digestibility of the protein, crude fiber, N-free ext. and ether ext., the cows rapidly became emaciated and the milk flow was affected. About 150 lbs. loss in wt. occurred. After the milk flow decreased, the digestibility of the protein and N-free extract increased, weight was gained, the rapidity of gain increasing as the lactation period passed. This decrease in digestibility resulted in a decrease in available net energy, until requirements for energy were just met, after which the decrease stopped. Excess protein as fed here seems to induce formation of milk fat from either protein or carbohydrates. A max. amt. of protein used for maintenance by the high-energy cows could not have been more than 0.345 lb. daily.

R. B. DEHMER.

**Heat production, basal metabolism and minimal metabolism of the tropical negro.** A. OZORIO DE ALMEIDA. *J. physiol. pathol. gén.* 18, 958-64 (1920); cf. de Almeida, *J. Physiol. Pathol. Gen.* 18, 712 (1920); *C. A.* 14, 2208.—Using the same technic as described in the earlier paper A. finds that the values for both the basal and min. metabolism of negroes inhabiting the tropical climates are approximately the same as are those for whites living in the same regions, the black color of the skin apparently not affecting heat elimination. The negroes also give evidence of a much lower metabolism than that which characterizes the inhabitants of temperate or cold climates. The mean value for the basal metabolism given by 47 detns. made on 20 subjects, 10 whites and 10 negroes, is 31.6 cal., which is about 20.4% less than that of the inhabitants of cold or temperate climates. This reduction is not an expression of a pathol. state, but is an indication of an adaptation to the higher temps.

F. S. HAMMETT

**The body weight and the nutritive conditions of the male charges of the Saxon State School at Colditz during the war years 1915-1919.** DEHIO. *Vierteljahrsschrift. ger. Med.* 60, 27-50 (1920).—Curves and tables are given showing the gradual loss of wt. from the beginning of 1915 to 1917 when the lowest level was reached. A slight betterment in conditions took place during the year 1917 and after the middle of 1918 gradual and continued recovery of body wt. is noted up to the termination of the reported period, March, 1920. The highest mortality occurred in the two years following the attainment of lowest wt. and was more than double that of the previous years. When food conditions improved in 1919 a general improvement in health and vitality was noted.

F. S. HAMMETT

**Dried milk powder in infant feeding.** W. H. PRICE. *Public Health Reports* 35, 809-28 (1920).—The lab. analyses as made in this study indicate that the dry milk powders and their remade products are safe for infant feeding.

J. H. L.

## F—PHYSIOLOGY

ANDREW HUNTER

**Physiology of blood sugar. III. The residual reduction of blood.** RICH. EGG. *Biochem. Z.* 107, 229-45(1920).—The reduction methods for the detn. of glucose in blood might be expected to yield too high results, because other blood constituents also reduce the soln. usually employed. This extra reduction over and above that represented by glucose is called the "residual reduction" and is detd. by the difference between the total reduction and the reduction after fermentation with yeast under conditions in which the auto-reduction of the yeast is detd. and the fermentation activity controlled. It was found in studies made of arterial and venous blood that the residual reduction is extraordinarily small, ranging from 0.000 to 0.008% in terms of glucose. Corpuscles and plasma yield figures of the same order. Cf. C. A. 12, 2361. F. S. H.

**Antagonism of concentration between the principal substances dissolved in the urine of man and the herbivora.** J. CHAUSSEIN. *J. physiol. path. gén.* 18, 895-957 (1920).—An extensive presentation of 5 series of observations concerning the concns. of urea and chlorides in the urine at successive intervals throughout the 24 hrs. of the day under conditions of regulated diet. There exists a remarkable coincidence between the weak concns. of the chlorides and high concns. of urea during sleep; while the nocturnal diminution of the urinary output, as a rule, indicates that during this period the urine is strongly concd. Moreover, during the morning period particularly (a period somewhat distant from the last meal), there occurs a urinary output but little variable and but slightly increased, the successive emissions of which show progressive diminution in urea concn., accompanied by successive increments of chloride content to the extent that the sum of the urea and chloride concns. tends to remain a constant. In fact, this phenomenon is characteristically continuous through the 24 hrs. and seems to be dominated by the chloride elimination rather than by that of the urea.

F. S. HAMMETT

**The peroxidase of milk, particularly the peroxidase of human milk.** A. B. MARFAN. *J. physiol. path. gén.* 18, 985-95(1920).—Studies on the peroxidase of human milk indicate that the reaction is often negative in older women (those over 35 years), in those cases where the milk flow is scanty and in cases where the nursing period has extended over a year. At times menstruation and pregnancy inhibit for a time its appearance in the milk. Tuberculosis and pleurisy appear to be able to suppress its reaction. In 9 out of 12 cases examd. the infants fed on milk lacking peroxidase showed evidence of malnutrition, which is taken to indicate that a deficient peroxidase secretion in the milk is a symptom of insufficiency of the nurse. F. S. HAMMETT

**Changes in the blood after the ingestion of food.** W. ARNOLDI. *Z. exp. Path. Ther.* 21, 97-103(1920).—Studies of the hemoglobin content of the blood at 10-min. periods for 2 hrs. or more after the ingestion of small amts. of food with or without liquid intake are given, together with the changes taking place in the HCl content of the gastric secretion. The results indicate to A. that there occurs a shifting of liquids between the tissues and blood. The stronger stimulators to gastric secretion usually cause a stronger reaction than do the weaker gastric stimulators. The moment of depression of the an- and subacidity finds a corollary in a drop in the hemoglobin curve. The serum protein concn. changes notably after the ingestion of a Ewald-Boas test meal. All the changes in concn. of the blood are attributed to vasomotor influences.

F. S. HAMMETT

**Digestion leucocytosis.** E. F. MULLER. *Z. exp. Path. Ther.* 21, 136-40(1920).—A discussion presenting no new data.

F. S. HAMMETT

**Have the amino acids merely a secretin character?** SCHWEITZER. *Biochem. Z.* 107, 256-67(1920).—Studies are reported of the amt. of gastric secretion produced under the influence of the oral, subcutaneous and intravenous administration of amino



acid mixts. and preps. from the hydrolysis of liver, casein, fish, and meats with HCl at 100°, from which S. concludes that the amino acids do not possess the general property of inducing gastric secretion, since it was the exception for him to obtain an increased secretion after the administration of the preps. mentioned. He is of the opinion that the carriers of the secretion stimuli which occasionally was observed with the amino acid mixts. are not the amino acids but other compds. originally present in the mother substances and which were liberated by the hydrolysis. F. S. H.

A note on the sterility of drawn blood. H. L. WHITE AND THOMAS WATSON. Univ. So. Calif. *J. Lab. Clin. Med.* 6, 45-6(1920).—Human and ox blood samples were analyzed in 3 portions, one immediately after withdrawal, the other two after varying periods at room temp. or in the ice box. Very little decomposition was noted, and negligible change occurred in non-protein and urea N, creatinine, uric acid and sugar in 20 hrs. at room temp. E. R. LONG

#### G—PATHOLOGY

H. GIDEON WELLS

Active immunization against diphtheria in a large child-caring institution. J. BLUM. Home for Hebrew Infants, New York. *Am. J. Diseases Children* 20, 22-8(1920).—The susceptibility of children to diphtheria was detd. by the Schick test. Those found susceptible, 252 in number, were immunized with toxin-antitoxin mixt. They received the Schick test at two-month intervals. After 3-4 months 184 (73%) gave negative Schick. Of the 130 cases observed 9-10 months 118 (91%) were negative, and of the 50 observed after 22 months all were negative. S. AMBERG

Nonspecific immunity. H. MUCH. Univ. Hamburg. *Deut. med. Wochschr.* 46, 483-4(1920).—Nonspecific immunity can be increased by the most different harmless substances foreign to the body. They stimulate the organism to defense and elimination. At the same time the general powers of defense against the disease-producing germs are stimulated. Treatment with properly balanced partial antigens will prove of greater advantage. These partial antigens are split protein, lipid and fat. The effect of most physical methods of treatment is ultimately due to the stimulation of the nonspecific immunity (treatment by light, rays, electricity, massage, baths, magnetism (sic!)). The treatment by forced feeding or fasting also belongs here. Some grotesque remarks are made about the nature of disease. S. AMBERG

Further remarks concerning nonspecific immunity. H. MUCH. *Deut. med. Wochschr.* 46, 791(1920).—Generalities. Cf. preceding abstr. S. AMBERG

The significance of the kidney function in diabetes mellitus. H. SCHIROKAUER. Univ. Berlin. *Deut. med. Wochschr.* 46, 791-3(1920).—Attention is called to those cases where a glucemia exists without glucosuria in the presence of nephritis. The possibility of an influence of an internal secretion of the kidneys is considered, preventing the accumulation of sugar in the tissues under such circumstances. S. A.

The use of the Abderhalden dialysis method in cases of renal tuberculosis. F. WAUSCHKUN. *Deut. med. Wochschr.* 46, 711-3(1920).—The Abderhalden method did not permit distinguishing a renal tuberculosis from acute or chronic nephritis. S. AMBERG

Coma diabeticum in pregnancy. F. UMBER. *Deut. med. Wochschr.* 46, 761-3(1920).—A woman pregnant six months had a diabetes belonging perhaps to the so-called renal type. The carbohydrate tolerance was very good. On a given diet the sugar disappeared from the urine while the amount of  $\text{NH}_3$  and  $\beta$ -hydroxybutyric acid increased. From a blood sugar level of 0.107% fatal coma developed quite suddenly. The glycogen content of liver and placenta was very small. The heart blood of the baby removed by operation immediately after its death contained 0.091% sugar. The baby died 24 hours after birth. S. AMBERG

Investigations with the lues reactions of Meinicke (M. R. and D. M.). O. BLASUS. *Deut. med. Wochschr.* 46, 854-6(1920).—M. R. is the, original Meinicke reaction, D. M. is the third modification. A large number of sera were tested. The Wassermann reaction did not work in about 4.5%, M. R. in about 4.3% and D. M. in about 2.5%. The question whether nonspecific reactions occur more frequently with the Meinicke reactions than with the Wassermann reactions has not yet been answered.

S. AMBERG

Immunization against tuberculosis with massive antigen dosage. UHLENHUTH AND JOEYTEN. *Deut. med. Wochschr.* 46, 877-9, 901-2(1920).—It is possible to sep. the wax of the tubercle bacilli from the protein with  $\text{CHCl}_3$ . Tubercle bacilli of different sources were used. Guinea pigs and rabbits were treated with bacilli from which the wax was removed, with the wax and with a combination of both. The dosage was large.

S. AMBERG

Can casein introduced parenterally produce a particularly strong protein action owing to its physical-chemical properties? R. RIEDEL. Univ. Freiburg. *Deut. med. Wochschr.* 46, 881-2(1920).—From the absence of glyccocoll and presence of relatively large amts. of tyrosine and tryptophane, as well as from the fact that casein is attacked by erepsin, it is assumed that it is easily decompd. in the organism. On intravenous injection erepsin is supposed to split off quickly, mono- and diamines stimulating cells to the activation of ferments. The high sp. rotation of casein (80-97.8 degrees) may play some role.

S. AMBERG

The solution of the tuberculin riddle. (Conclusion of the partigen treatment.) H. MUCH. Univ. Hamburg. *Deut. med. Wochschr.* 46, 845-6(1920).—Any mixt. of reactive substances can be divided by treatment with acid into a water-sol. and a water-insol. partigen (partial antigen). The water-insol. partigen can be divided into protein, lipid and neutral fat partigen. Best known are those from the tubercle bacillus. The water-sol. part *L* is the pure tuberculin. The residue *R* is composed of (a) protein, *A*, (b) fatty acid-lipoid, *F*, (c) neutral fat—fat alcohol, *N*. All of these call forth local or general manifestations in man, but the infiltrations differ in number and kind of cells. The *L* hypersensitiveness is a toxin hypersensitiveness which can disappear on repeated injections of *L*. The *R* hypersensitiveness is an immune-hypersensitiveness. Tuberculous guinea pigs succumb to injections of *L* but not of *R*. The tuberculins of commerce contain *L* and *R*. The first partigen law is: Every disease microorganism can be divided in partial antigens and each partial antigen has a sp. antibody. The second law is: A test cannot be made with the combination of all partial antigens. A reaction may be produced to one or the other antigen, while antibodies to all the partial antigens are not present. Total immunity can only be detd. by the use of the partial antigens. The third law is: A combination of partial antigens may not elicit a reaction because the partial antigens may influence each other. Separation into the several partial antigens may therefore activate the partial antigens. These laws are applied to the treatment of tuberculosis. If possible, treatment with the three *R* antigens is started. This produces a hypersensitiveness to these antigens, which is useful. At the same time the hypersensitiveness to *L* (harmful) usually diminishes. It may be necessary to stop the treatment with *A*, *F* and *N*, and reduce first this hypersensitiveness by treatment with *L*. Stimulation of the nonspecific immunity can be evoked at the same time by various methods of treatment. Cases of phlyctenular conjunctivitis are unfavorably influenced by the *R* antigens. They show marked hypersensitiveness to *L*. If this is overcome the disease heals.

S. A.

Examination of stomach contents of children. H. BRÜNNING. Univ. Rostock. *Deut. med. Wochschr.* 46, 883-4(1920).—Children, 300 in number, 2-15 years of age, received a test breakfast of rolls and tea, the test meal being removed 45-60 min. later. The total acidity and the free  $\text{HCl}$  were detd., but not the  $\text{HCl}$  deficit. The av. value

for total acidity was 48.4 cc. 0.1 *N* acid, for free HCl 14.3 cc. From 2 to 10 years the total acidity was 46.9, free HCl 11.5; from 11 to 15 years total acidity was 52.8, free HCl 20.1. In cases of gastro-intestinal disorders total acidity and free HCl were diminished. .

S. AMBERG

**Biochemical findings in a case of hemolytic icterus with splenomegaly.** W. SCHOEN. *Deut. med. Wochschr.* 46, 908-10(1920).—The patient was examd. in two attacks. In one the red blood corpuscles were dissolved by 0.9, in the other by 0.8% NaCl soln.; later when the anemia had improved it took 0.6% soln.; even this is abnormal. Own serum as well as serum of other individuals acted hemolytic; with increase of the number of erythrocytes and of the hemoglobin this was no longer the case. The serum of the patient was not hemolytic to other erythrocytes. The bilirubin content of the blood and the urobilin of urine and feces were increased. Tests of the liver function with galactose and levulose did not show anything abnormal. The urine at first dark brown, did not contain any bilirubin, but some urobilin and much urobilinogen. The patient reacted to 1 mg. adrenaline given subcutaneously with severe collapse and later severe headache. Free and combined cholesterol of the blood was increased after the adrenaline injection, but not the bilirubin.

S. AMBERG

**Depression immunity. Superinfection with chicken cholera bacilli.** BERLINER AND CITRON. Univ. Berlin. *Deut. med. Wochschr.* 46, 997-8(1920).—A culture of chicken cholera bacilli was fatal to guinea pigs in doses of 1/1000000 of 1 cc. of a 24-broth culture. On intraperitoneal injection death occurred the next day. On subcutaneous injection of 1/100 cc. death occurred after a week or more. Guinea pigs having received the latter dose subcutaneously were injected the day after intraperitoneally with 1/5000 cc. These animals lived 6-8 days. With increase of the second dose to 1/1000 cc. and more death occurred in a day.

S. AMBERG

**The complement-fixation reaction of Dietrich in fever.** A. MAYER. *Deut. med. Wochschr.* 46, 998-9(1920).—Dietrich described a complement-fixation reaction of the blood of fever patients using iodothylin as antigen. The antigenic property is due to the presence of an organic thyroid substance in iodothylin. This reaction was found positive in all cases with increased body temperature with a few exceptions. In some cases of typhoid fever, pneumonia and Weil's disease it was negative. But all of these negative cases were soldiers who had more or less severe myxedema. The same individuals tested with intramuscular injections of 5 cc. milk during convalescence did not react with a rise of temperature or with very little. The Dietrich reaction is positive in fever due to various causes. In cases of athyreosis it is negative, in cases of thyreotoxicosis it is positive even when free from fever.

S. AMBERG

**The sessil receptors in anaphylaxis and the role of the autonomous nervous system in the anaphylactic symptom complex.** W. ARNOLDI AND E. LESCHKE. Univ. Berlin. *Deut. med. Wochschr.* 46, 1018-9(1920).—Expts. were made on sensitized frogs using the Trendelenburg arrangement (perfusion of lower extremities through aorta and registration of the drops escaping from the abdominal vein). First Ringer soln. was perfused for 24 hours, all blood and lymph being removed. On addition of antigen the vessels dilated, while in the controls they contracted. This proves sessil receptors. Anaphylatoxin prepared *in vitro* had the same effect. It is thought that the symptoms of the anaphylactic intoxication can be explained in its essentials by an excitation of the endings of the parasympathetic nervous system in the smooth muscles.

S. AMBERG

**The theory of the lipid-combining reaction.** F. MEINICKE. *Deut. med. Wochschr.* 46, 1022-3(1920); cf. *C. A.* 14, 2372.—Theoretical considerations of the author's M. R. lues reaction.

S. AMBERG

**The antipoisonous action of lecithin.** H. LEO. Univ. Bonn. *Deut. med. Wochschr.* 46, 1045-6(1920).—Lecithin *ex ovo purissimum* (Merck) was used in the

expts. as a 1-2% emulsion in physiol. salt soln. prepared by shaking for days. Injected intravenously in rabbits it increases the respiratory ventilation. It had no or little influence on ether or  $\text{CHCl}_3$  narcosis, but it does counteract the diminution of the respiratory ventilation brought about by morphine. Camphor alone increases the ventilation about 200%; lecithin alone about 40%. Simultaneous administration of camphor and lecithin increases it only about 15%. Attention is called to the possibility that lipemia in man may interfere with morphine action.

S. AMBERG

Toxins and antitoxins of the cholera bacilli and other similar bacilli. The question of the endo- and anti-endotoxins. R. KRAUS. Hyg. Inst., Buenos Aires. *Deut. med. Wochschr.* 46, 1046-8 (1920).—Immune serum obtained with vibrio cholera toxin contains sp. agglutinins, precipitins and bacteriolysin for itself and for the *Vibrio El Tor*, but it contains antitoxin only against vibrio cholera toxin. With this serum mice can be protected against cholera but not against *El Tor*. *El Tor* immune serum also contains antitoxin against cholera toxin. The toxins of *Vibrio Nasik* yield an immune serum which is antitoxic against the toxins of *Nasik* and *El Tor*, but not against cholera. It is bacteriolytic only for *Nasik*. Cholera serum does not protect against *Nasik*. Only antitoxic and not bacteriolytic serum can protect. The toxins of the cholera bacillus and not the endotoxins produce cholera. Anaphylatoxin could only be prepared from guinea pig and rat serum, not from rabbit, dog, or horse serum. It is only toxic for guinea pigs.

S. AMBERG

A note on renal function in scarlet fever. B. S. VEEDER AND M. R. JOHNSTON. Washington Univ. *Am. J. Diseases Children* 19, 223-8 (1920).—The kidney function was tested with phenolsulphonaphthalein and by the detn. of the non-protein N of the blood. The albumin appeared in the urine before changes in renal function took place.

S. AMBERG

Precipitin for egg albumin in stools. C. G. GRULEE. McCormick Mem. Lab. *Am. J. Diseases Children* 20, 15-7 (1920).—Stools of 24 patients, 2 years of age or over, were examd. with an anti-egg white rabbit serum of a precipitin titer of 1:40000. Of the 103 stools 3 showed positive reactions. Of 17 stools from 3 cases, 13-22 months of age, only one showed a doubtful reaction on the administration of 1 teaspoonful of egg albumin each day. The third group of cases were infants under 2 years of age. Of the 33 cases only two were more than 1 year old. Many were new-born infants who had received a dilute albumin water. Five of 221 stools gave positive reactions. One of these occurred in a new-born infant, who had been given egg albumin water. Three other children gave positive reactions, which cannot be accounted for. S. A.

Senecio disease, or cirrhosis of the liver caused by Senecio poisoning. FREDERICK C. WILMOT AND GEORGE W. ROBERTSON. South Africa. *Lancet* 1920, II, 848-9. —A disease in human beings resembling Pictou disease of cattle due to the ingestion of *Senecio ilicifolius* and *S. burchelli*. Seeds and portions of these weeds remain mixed with wheat during threshing. The symptoms are those of an acute gastro-intestinal disturbance with ascites. Autopsy findings revealed cirrhosis of the liver. The disease was reproduced in guinea pigs and rats by exptl. feeding.

E. B. FINK

Coeliac infantilism; its fat digestion and treatment by bile salts. REGINALD MILLER, JOHN WEBSTER AND HERBERT PERKINS. *Lancet* 1920, II, 894-7.—Coeliac disease is characterized by abdominal distention without organic disease, persistent or intermittent diarrhea with pale fatty stools and retarded physical growth and development. In coeliac disease there seems to be no interference with fat-splitting since fatty acids occur in normal proportions. The excess fat in the stool is apparently due to deficient absorption of fatty acids. No disturbance in pancreatic function could be demonstrated. The administration of bile salts tended to diminish the % of fat excreted in the feces. It is suggested that the condition may be due to diminished excretion of bile salts into the intestine.

E. B. FINK

**Variations in albumin content of the cerebrospinal fluid in the psychoses.** P. RAVAUT AND LAIGNEL-LAVASTINE. *Bull. mem. soc. med. hop.* 36, 1151-7(1920).—In dementia precox cases the protein of the cerebrospinal fluid is remarkably constant, ranging closely around the normal values. Similarly normal figs. were obtained with chronic systematic hallucinated patients. Among the melancholics the amounts vary within the range of 0.025 to 0.040. In epileptics the albumin values are variable in the individual as well as in the group and usually high. Similar findings were obtained in imbeciles, mutes, etc. Since higher values than normal are found in these several types of mental disturbance while the cell count remains normal the authors tentatively admit the existence of a disturbance of the central nervous system or its membranes.

F. S. HAMMETT

**The action of extracts of the glands of internal secretion in osteomalacia.** A. ELFER AND J. KAPPEL. *Z. exp. Path. Ther.* 21, 104-27(1920).—An extensive series of expts. on one patient in which the N, Ca, Mg and P metabolism was studied with and without the administration of products of the pituitary, thymus, adrenals and ovary. The authors do not venture to draw any conclusions from the study but note that no P loss occurred during the 149 days of the expt., while a negative Ca and Mg balance occasionally did occur.

F. S. HAMMETT

**The polypeptide and amino-acid portion of the non-protein nitrogen of the blood.** F. SCHWERINER. *Z. exp. Path. Ther.* 21, 129-35(1920).—Using the formol titration method for the detn. of the amino acids of the blood in the filtrate obtained by dilg. 100 cc. blood with 100 cc. H<sub>2</sub>O and pptg. the proteins with 200 cc. 2% HCl and 200 cc. 5% HgCl<sub>2</sub> soln., S. obtained a series of values which, taken together with his results for the total non-protein N and the peptide N lead him to conclude that the majority of deproteinized bloods from the commonly encountered diseases do not contain polypeptide N and that the amino-acid N represents 9.3% of the total. In the tumor cases studied polypeptides were found but the amino-acid content remained within normal values. In disorders of the liver similar results were found in some cases. In cases of kidney disease the amino-acid N is increased.

F. S. HAMMETT

**Anaphylatoxin, characterized by a precipitation phase of the serum globulin.** HERMANN DOLD. *Arch. Hyg.* 89, 101-29(1920).—From a study of the experimental data in the literature it is decided that the formation of anaphylatoxin is dependent on the surface and especially the quality of the surface of the agent added to fresh serum. The addition of bacteria, which are known as good anaphylatoxin formers, to fresh serum (not inactive serum) produces a cloudiness which is a constant manifestation and is related to the formation of toxin. The usual explanations for this are unsatisfactory. By the use of the microscope with the Tyndall illumination (seroscopy) it is possible to see the process of formation. By this method of study it can be shown that in anaphylatoxin-containing sera there is a peculiar state of flocculation which develops after the addition of agents which are formers of anaphylatoxin. It goes over, sooner or later, into a definite ppt. The course of this flocculation process is chiefly dependent on (a) the freshness (activity) of the serum; (b) species of serum; (c) on the properties of the added agents; (d) the quantitative relations between serum and agent; (e) the temp.; (f) the presence and amt. of sp. amboceptor. By the addition of salts (especially Na acetate), there is a stabilization of the flocculation and conservation of toxic action. The significance of seroscopic observation for the comprehension of the mechanism of anaphylatoxin action, active and passive anaphylaxis, primary serum toxicity, serum disease and drug idiosyncrasies is discussed.

JULIAN H. LEWIS

**The increase of antibody on intravenous injection of methylene blue.** TH. FÜRST. *Arch. Hyg.* 89, 161-7(1920).—While there are some substances which on increasing the amt. of antibody formation, also increase the no. of leucocytes, there are others which increase antibodies and at the same time produce a leucopenia. Methylene blue

belongs to the latter class. It acts probably by injuring certain cells whereby antibody is set free. For this view is the evidence that the antibody increase is short-lived and its maximum is reached when the leucocyte count is minimal. JULIAN H. LEWIS

**The phenolsulfonephthalein test and the non-protein nitrogen of the blood in chronic nephritis.** REGINALD FITZ. *Bost. Med. Surg. J.* 83, 247-54(1920).—The phenolsulfonephthalein test and the non-protein N concn. of the blood are not of obvious value in the diagnosis of chronic nephritis as they do not point out the presence of any sp. pathological type of lesion in the kidney and they do not demonstrate the presence of kidney disease in the absence of common physical signs. From a pathological point of view, there are 2 common types of chronic nephritis, chronic glomerulonephritis and arteriosclerotic nephritis. The differentiation of these diseases cannot be made by detg. renal function. As the lesions of chronic nephritis advance, the phenolsulfonephthalein excretion diminishes and the non-protein N concn. of the blood increases. At present, however, a single observation with these tests gives less prognostic information than does careful clinical examinf. JULIAN H. LEWIS

**The significance of the lipoids in the resistance to tuberculosis.** J. DE SIKAS PALMA. *Centr. Bakt. Parasitenk., Abt. I* 83, 231-54(1919).—The lipoids from the lymph glands of tuberculous cows, dissolved in olive oil or emulsified in bile, produce necrosis and edema when injected subcutaneously. The injected animals may die. The unsatd. soaps of the lipoids from normal mesenteric glands of tuberculous cows are toxic. Analyses of the mesenteric glands and pancreas showed a certain contrast between these glands and their relation to tuberculosis, in that the free lipid-acids of the mesenteric glands of tuberculous cows have more unsatd. groups than their mother substance and the analogous acids from normal glands. In the pancreas the reverse was found. The mesenteric glands of tuberculous cows probably contain a poisonous compd. from the tubercle bacilli because of a deficiency of fat-splitting ferments which are plentiful in the pancreas. The soap-fraction of the pancreas of tuberculous cows is never toxic; it contains no substances which give a ninhydrin reaction. For this reason the lymph glands are foci of tuberculous infection, while the pancreas is never found tuberculous. The lipoids from tuberculous animals differ from those of healthy animals. They have a lower m. p., are darker in color and become brown when heated. The acidity of these preps. is demonstrated in the fact that they promote the solution and phagocytosis of tubercle bacilli and that they produce myelin figures and a condition of easy bacteriolysis in isolated tubercle bacilli. This activity seems to be dependent on the presence of unsatd. fatty acids because it is lowered by satn. with I. Pancreatic juice stimulates the phagocytic action of leucocytes. The satd. soaps from the pancreas of tuberculous cows are inactive when put on the conjunctiva while the unsatd. soaps produce a severe inflammation when injected subcutaneously into the guinea pig. Also the pancreatic satd. soaps are innocuous when injected into man. J. H. L.

**The theory of the serology of typhus fever blood and etiologic significance of the X-strain of the proteus bacillus.** EMIL EPSTEIN. *Centr. Bakt. Parasitenk., Abt. I* 83, 255-81(1919).—The explanation of the various serum reactions in typhus, as the Weltmann pptn. reaction, the Weil-Felix agglutination, the agglutination of other bacteria from typhus cases, the Wassermann reaction and the complement fixation reaction with bacterial antigens, is the changed physical condition of the typhus serum which leads to an increased tendency for it to be pptd. The Weltmann reaction is the direct outcome of this condition while the complement fixation and the Weil-Felix reactions are the indirect result. The X-strain of proteus has no relation to the etiology of typhus. Its agglutination in spotted fever depends on secondarily acquired properties. JULIAN H. LEWIS

**The action of cholera and typhoid vaccines prepared by different methods on the agglutination titer.** STANISLAW SIERAKOWSKI. *Centr. Bakt. Parasitenk., Abt. I* 84,

161-78(1920).—If judged by the results with a large group of people, the method of prepn. of the vaccines has a great influence on the agglutination titer. The substances used favorably in the prepn. of typhoid vaccine (CHCl<sub>3</sub> and formalin) may be disadvantageous with cholera vaccines. On account of the ease of contamination the use of sterilization by heat is limited. Such vaccines must be preserved with disinfectants, and if disinfectants must be used, heat is superfluous. Glycerol can be used to kill cholera vibrios but not for preserving them. Heated cholera vaccines produce a smaller no. of agglutinins in the blood. The agglutination produced after 2 injections of cholera vaccine is no more than after 1 injection. After either 1 or 2 injections the agglutinins will have disappeared from the blood. Sensitized typhoid vaccines give no different results than nonsensitized vaccines. Typhoid vaccines made from any single strain give the same results except from a 10 yr. old museum strain. JULIAN H. LEWIS

**The serodiagnosis of glanders in asses and mules.** W. PFEILER AND FR. GRÄFF. *Centr. Bakt. Parasitenk., Abt. I* 84, 279-87(1920).—The presence of anticomplementary substances, in the serum of mules and asses is not constant and therefore no relationship between it and the phylogenetic relation of the 2 species of animals can be established. Nonsp. fixation of complement by a given serum makes the serodiagnosis of glanders difficult. JULIAN H. LEWIS

**The action of normal serum in anthrax infection.** FRANZ GERLACH. *Centr. Bakt. Parasitenk., Abt. I* 84, 396-400(1920).—The claim of Kranz and his co-workers that normal cattle, horse and sheep sera gave protection against infection with anthrax could not be confirmed. JULIAN H. LEWIS

**The relation of the blood sugar on high mountains in normal and pathological conditions, and after exposing to heat, rays and the sun.** H. C. FRENKEL-TISSOT. *Deut. Arch. klin. Med.* 133, 286-305(1920).—Healthy people in the mountains show the same blood-sugar relations as on the plains. In internal and surgical diseases especially in disease of internal secretion the blood sugar is the same as in patients with the same diseases on the plains. However, in normal people on the mountains, sunning, raying with the quartz-lamp and hyperthermia have a very different action. Those with a high blood-sugar value have a tendency to be lowered by these effects while those with normal or low values have a tendency to be increased. JULIAN H. LEWIS

**The change of blood alkalescence in disease.** BERNHARD KUHLMANN. *Deut. Arch. klin. Med.* 133, 346-63(1920).—The acrotonometric method which has been developed by J. Barcroft, Morawitz-Walker and Münzer-Neumann, appears to be the best method for giving an idea of the disarrangement of the acid-base equil. of the blood. It has several advantages over the usually used alveolar air method of Haldane which are described in the text but it has the disadvantage that one must work in man with venous blood more from a single area. In decompensated heart disease and in lung diseases, in which there is a dyspnoea, there is only a moderate decrease of the blood alkalescence. According to present notions one can decide that only seldom is there a greater deficiency of O. In anemia, there is also only a slight decrease in blood alkalescence. Higher grades of decreased blood alkalescence are seen in febrile diseases, in diabetes with acidosis and in kidney deficiencies. In these conditions there is probably not only a diminished supply of O but also an abnormal process of intermediary metabolism. JULIAN H. LEWIS

**Chemical changes in the blood in disease. VII. Chlorides.** VICTOR C. MYERS. *N. Y. Post Grad. Med. School and Hosp. J. Lab. Clin. Med.* 6, 17-22(1920); cf. *C. A.* 14, 1577, 1711, 2819, 2820, 3458.—The normal plasma Cl calculated as NaCl varies between 0.57-0.62%, the threshold value being quite constant at 0.562%. The whole blood value is 0.45-0.50%. Measurement of Cl in plasma should be made on blood very soon after withdrawal, for with loss of CO<sub>2</sub> on standing Cl passes from the corpuscles. Blood Cl is increased in nephritis, heart disease with passive hyperemia,

anemia, and malignancy, while low values have been found chiefly in fevers, diabetes and pneumonia. Blood Cl detn. in nephritis is an excellent check on the extent of Cl restriction required. The Cl retention is much more marked in parenchymatous than in interstitial nephritis. The Cl retention of pneumonia is associated with a decreased Cl content of the blood. The most convenient means of detn. is the Volhard method, following pptn. of the blood proteins with picric acid in the creatinine and sugar estn.

E. R. LONG

**Development of the bactericidal power of whole blood and anti-bodies in serum.** J. H. BLACK, K. FOWLER AND P. PIERCE. *Baylor Univ. J. Am. Med. Assoc.* **75**, 915-9(1920).—Conclusions: The bactericidal power of blood as detd. by Heist and Solis-Cohen (*C. A.* **13**, 461, 3229) is the most dependable criterion of the actual immunity of the animal. Development of the bactericidal power of the blood against typhoid and Shiga bacilli was practically identical with that of the serum. The route of inoculation made no material difference in the rapidity or height of the development of bactericidal power. The agglutinins and complement-fixing bodies were only roughly comparable to the bactericidal power. The leucocyte counts and phagocytic index were of no value in detg. the degree of immunity. In the rabbit immunized to typhoid and dysentery bacilli, lysis occurs with great rapidity, and a short incubation period is sufficient. The mechanism of lysis is obscure. Phagocytosis probably was not a factor. Citrating and defibrinating blood of rabbits immunized to typhoid and dysentery bacilli did not affect the bactericidal activity save to slow the reaction. In the blood of typhoid and dysentery immune animals, contaminating organisms may grow luxuriantly. A short incubation period removes most of the difficulty due to contaminants. The refractory state of Teague and McWilliams is shown to depend probably on a rapid rise in bactericidal power, and the rapidity of mobilization of this power varies with the route of injection. Inactivation of serum of immunized rabbits did not materially reduce the bactericidal action.

L. W. RIGGS

**A study of the oxidase reaction with  $\alpha$ -naphthol and  $p$ -phenylenediamine in tumors.** MAUD L. MENTEN. *Univ. Pittsburgh. J. Cancer Res.* **6**, 321-44(1920); cf. *C. A.* **14**, 564.—M. shows that a development in color in the reagent is due to oxidation so when the adsorption of the reagent by a tissue is followed by an increase in intensity of the color, increased oxidative capacity has been developed in the reagent, and a corresponding tissue reduction. The more marked the oxidase reaction the greater the reduction in the reacting tissue and *vice versa*. Cell constituents of certain tissues in which a slight oxidase reaction normally develops, may, through degenerative processes, be converted into new colloidal complexes in which a max. reaction obtains. Examples of such alterations in intensity of reaction are found in connective tissue (spindle-cell sarcoma) and in stratified epithelium (epithelial pearls of epithelioma). These variations are associated with a transformation of potential into kinetic energy and indicate a considerable degree of lability of granules. This lability of cell granules occurs in tissue which has a high regenerative capacity as measured by variations on the oxidase reaction. In the nuclei intensity of color may be increased or diminished, the former in degenerative processes, the latter in rapidly proliferating cells.

E. P. WOLF

**The peroxidase reaction in three cases of multiple myeloma of the bones with remarks concerning the nosological position of these tumors.** PLINN F. MORSE. *Harper Hosp., Detroit. J. Cancer Res.* **5**, 345-71(1920).

E. P. WOLF

#### H—PHARMACOLOGY

ALFRED N. RICHARDS

**Synthetic dyestuffs as applied in chemico-therapy.** GEORGE HEYL. *Urological and Cutaneous Rev.* **24**, 320-5(1920).—A review.

H. V. ATKINSON



The pharmacological action of *Adonis vernalis*. JAMES M. SCHMIDT. *Am. J. Pharm.* 92, 702-14(1920).—S. concludes that *Adonis vernalis* stimulates the heart in a manner very similar to digitalis; that it produces variations in blood pressure characterized by a rise, if large doses are administered, or by no pressure effect if the doses are relatively small; that the changes in pressure are caused mainly by the action of the drug upon the blood vessels; that it is less irritating than digitalis and possesses less tendency to produce a gastric disturbance; and that it possesses approx. the same strength as digitalis preps. made according to the official directions for a tincture or a fluidext.

W. G. GAESSLER

Toxicity and chemical potential. W. LASH MILLER. Univ. Toronto. *J. Phys. Chem.* 24, 562-9(1920).—Expts. were carried out largely on yeast cells to compare the toxicity of a disinfectant, as phenol in aq. soln., when mixed with an indifferent salt, as NaCl. The results are summarized in the statement that the efficiencies of such solns. depend on the chem. potential: Two solns. have the same toxicity if they are such as to be in equil. with the same soln. of phenol in an immiscible solvent. Some exceptions and complications are noted in carrying out the details which are given in full for several organisms and several salts other than NaCl. H. C. HAMILTON

Toxicity towards anthrax and staphylococcus of solutions containing phenol and sodium chloride. J. S. LEMON. Univ. Toronto. *J. Phys. Chem.* 24, 570-84(1920).—On the principle established by Miller (preceding abstract) expts. were carried out on a number of spore-bearing organisms varying the conditions as to concns. of solns. and temp. of application. Results are included which, with some exceptions, seem to verify the conclusion that the increased toxicity of a phenol soln. on adding salt to it, is due to the increased potential. Solns. with and without salt are equally toxic if their compns. are such that both would be in equil. with the same soln. of phenol in toluene.

H. C. HAMILTON.

Bismuth subnitrate poisoning. AL. IONĂSCU. *Bul. soc. chim. Romania* 1, 80-4 (1919).—Since  $\text{BiONO}_3$  is an insol. salt it might be regarded as harmless ordinarily, but when the stomach fluids are distinctly acid and the intestinal contents alk.,  $\text{BiONO}_3$  is decompd., setting free the easily absorbable acid radical, especially when the mucous membranes are hyper-irritable.  $\text{BiONO}_3$  must always be regarded as a toxic salt, this action, however, being evident only from large doses and when the functions of the secretions of the digestive tract are over stimulated. Toxic action is nearly always caused by the  $\text{NO}_2$  radical which, when reduced in the small intestine and absorbed by the blood in the form of  $\text{HNO}_2$ , brings about oxidation and other color changes. The manner in which these products are formed must be explained not only by the action of  $\text{NO}$ , formed by the dissociation of  $\text{HNO}_3$ , but also by the action of hyponitrite and of  $\text{HNO}_2$  on the protein substances and on the coloring matter of the blood. On the formation of these products further expts. are to be carried out. H. C. H.

The action of the corrosive esters: particularly the war gases. C. WACHTEL. *Z. exp. Path. Ther.* 21, 1-18(1920).—Studies with kymograph tracings of the effects of various esters on the blood pressure and respiration of rabbits showed that the functional alterations which are induced by the alkyl esters of the mineral acids, of  $\text{H}_2\text{CO}_3$ , and the org. acids studied consist of a slower and shallower breathing accompanied by a pronounced drop in blood pressure. Convulsions are caused by the action of the esters of the mineral acids on the nervous system, while the  $\text{H}_2\text{CO}_3$  esters and those of the org. acids bring on a more or less variable narcosis. When  $\text{Me}_2\text{SO}_4$ ,  $\text{Et}_2\text{SO}_4$  or  $\text{Et}$  bromoacetate are inhaled 3 typical forms of response are elicited.

F. S. HAMMETT

The opium problem. The action of opium and its derivatives on the intestine. FR. UHLMANN AND J. ABELIN. *Z. exp. Path. Ther.* 21, 58-74(1920).—(I) Since it was found that opium and its derivatives did not act exactly alike on the isolated and

*in situ* intestinal segments of warm blooded animals, an app. was developed, which is described, for studying the response of the intestine in the living animal to the drugs. The circular and longitudinal musculature of the small intestine are qualitatively similarly affected although the quantitative reaction is different. (II) In small doses opium retards peristalsis, in large, it acts as a stimulus, and in very large amounts produces 1st a max. stimulation and then inhibition. The pendular movements of the isolated segment can only be influenced by large doses and they are retarded. A bibliography of 314 titles is appended.

F. S. HAMMETT

Reactions following intravenous administration of arsphenamine. A. A. STRICKLER, *et al.* *N. Y. Med. J.* 112, 498-9(1920).—The results of this investigation indicate that the injection of one seventy-fifth of a grain of atropine sulfate or of 0.5 cc. adrenaline chloride (1:1000) previous to arsphenamine injections in no wise influences the occurrence of the usual early reactive phenomena.

F. S. HAMMETT

Action of nicotine on the dog heart. A. CLERC AND C. PRIZI. *J. physiol. pathol. gén.* 18, 965-75(1920).—Electrocardiographic studies of the effect of nicotine on the dog's heart show an acceleration of the autonomous rhythm of the ventricles and a series of abnormal centers concerned with the origin of the stimulus.

F. S. HAMMETT

Typhoid and anti-typhoid vaccination. F. RATHERY AND P. MATHIEU. *J. physiol. path. gén.* 18, 996-1013(1920).—Two pieces of work presenting data showing that preventative vaccination, while it may not absolutely protect from typhoid fever or typhic affections, does create in the individual a condition of quite special immunization which causes the typhic affection to develop in quite a different fashion. No clinical type can be individualized as "typhoid vaccinated."

F. S. HAMMETT

Poisoning by methyl bromide. F. ROHRER. *Vierteljahr. ger. Med.* 60, 51-9(1920).—Clinical description of several cases of poisoning by  $\text{CH}_3\text{Br}$ , among the symptoms of which are numbness and giddiness. Later effects seem to indicate a disturbance of the permeability of the membranes, and the cell respiration, which primarily is due to the  $\text{CH}_3\text{Br}$  action on the chem. mechanisms of the cells. The initial point of attack is not known, *i. e.*, whether the central nervous system or the endocrine glands are attacked, and thus secondarily disturb the organic functions as a whole. The possibility of a general alteration of intermediary metabolism is considered. The particular part of the mol. involved in the production of the toxic effects is also unknown, though the tendency is to attribute the action to the Me group.

F. S. HAMMETT

Poisoning by methyl bromide and detection of the compound in the blood and organs of poisoned animals. W. LÖFFLER AND W. RÜTMEYER. *Vierteljahr. ger. Med.* 60, 60-7(1920).—Rather complete clinical report of a case of fatal poisoning by  $\text{CH}_3\text{Br}$ . Analysis of the fresh blood and urine before death showed no traces of the toxic compd. nor could any be found in the brain or liver at autopsy. However, when guinea pigs were poisoned with  $\text{CH}_3\text{Br}$  until they died very soon after, positive reactions were usually found in the tissues examd. When the animals were so poisoned that they lived and *breathed* for some time after the administration of the vapor no traces of the  $\text{CH}_3\text{Br}$  could be found in the tissues.

F. S. HAMMETT

The treatment of leprosy with especial reference to some new chaulmoogra oil derivatives. J. T. McDONALD AND A. L. DEAN. *Pub. Health Rpts.* 35, 1959-74(1920).—The intramuscular injection of the ethyl esters of the fatty acids of chaulmoogra oil usually leads to a rapid improvement in the clinical symptoms of leprosy. In many cases the lesions disappear except for scars and permanent injuries, and the leprosy bacillus can no longer be demonstrated. When combined with I, the fatty acids of chaulmoogra oil and their esters give good results; but there is no adequate exptl. proof that this addition of I causes any increase in the effectiveness of the material

used. All the available evidence obtained from the use of fractions of the fatty acids of chaulmoogra oil indicates that the therapeutic action is due to one or more of the fatty acids of the oil or to some as yet unidentified substance associated therewith. The various methods of fractionation heretofore employed have failed to demonstrate the active agent. Although conclusive evidence is not at hand, it is probable that the oral administration of chaulmoogra oil derivatives is of minor importance compared with the injections. In treating leprosy, it is important to make use of all auxiliary agencies to build up and maintain bodily vigor. Hypodermic injections of the ethyl esters into leprosy nodules are followed by marked swelling with ultimate recession of the lesions. This is a valuable auxiliary treatment for especially resistant lesions.

JULIAN H. LEWIS

An experimental investigation of certain features of the pharmacological action of salvarsan. D. E. JACKSON AND G. RAAP. Univ. Cincinnati. *J. Lab. Clin. Med.* 6, 1-16(1920).—First class preparations of salvarsan have almost no direct action on the bronchial musculature of the dog. Small injections do raise the pulmonary blood pressure. This is counteracted by injection of adrenaline, probably because of a mechanical shifting of blood from the action of adrenaline on the systemic vasculature. The rise in pulmonary pressure is greater when the injection is made by way of the femoral vein than when made through the femoral artery. When the injection is made in the portal vein very little effect on pulmonary pressure is noted, a pptn. of the drug apparently occurring in the capillaries and arterioles of the liver. Some portion of the drug, however, must pass through, for fall in the carotid pressure occurred under these conditions.

E. R. LONG

Action of drugs on the output of epinephrine from the adrenals. VI. Atropine; pilocarpine. G. N. STEWART AND J. M. ROGOFF. Western Reserve Univ. *J. Pharmacol.* 16, 71-107(1920); cf. *C. A.* 14, 1156.—The rate of output of epinephrine from the adrenals (in cats) is not materially influenced by the action of atropine or pilocarpine. A moderate increase in the rate of liberation may be produced by atropine, which in large doses may be preceded by a moderate transient depression of output. The augmentation of the output, if any is caused, by pilocarpine, is small and is not comparable with the large increase in the rate of epinephrine liberation caused by strychnine or the immediate effect of nicotine.

C. J. WEST

Study of the action of cocaine on the splanchnic and cervical sympathetic neuromuscular mechanisms. ARTHUR L. TATUM. Univ. Chicago. *J. Pharmacol.* 16, 109-23(1920).—Cocaine intravenously administered greatly intensifies the blood pressure after stimulation of the peripheral end of a divided splanchnic nerve. This greatly increased response is largely due to the synergism between cocaine and the epinephrine secretion subsequent to the splanchnic nerve stimulation. In the absence of adrenal glands cocaine still increases the effects of splanchnic stimulation. Cocaine increases the constrictor effects of electrical stimulation of the cervical sympathetic nerves.

C. J. WEST

Action of borax on the uterus. J. W. C. GUNN. Univ. Cape Town. *J. Pharmacol.* 16, 135-9(1920).—Borax has a direct stimulant action on the uterus in concns. corresponding to toxic doses. This action is largely due to the alkalinity, as neutral solns. have less effect.

C. J. WEST

Toxicological study of some alcohols, with especial reference to isomers. DAVID I. MACHY. Johns Hopkins Univ. *J. Pharmacol.* 16, 1-10(1920).—The toxicity of the normal alcs., MeOH, EtOH, BuOH, AmOH—increases with their place in the aliphatic series, as indicated by the lethal dosage for cats, and by their effects on isolated frogs' hearts and plain muscle preps. The sec. Pr, Bu, and Am alcs. were found to be less toxic than the corresponding primary alcs. In discussing the toxicity of alcs., a dis-

tion should be made between the acute or immediate, and the secondary or remote effects of the drugs, as is well illustrated in the studies on MeOH and PhCH<sub>2</sub>OH.

C. J. WEST

**Further pharmacologic studies on arsphenamine.** MAURICE I. SMITH. U. S. Public Health Service. *J. Pharmacol.* 15, 279-95(1920); cf. C. A. 13, 2713.—The rise in pulmonary pressure produced by intravenous injections of arsphenamine is shown to be due to an obstruction of the pulmonary vessels, which may be due to mechanical blocking or to a constricting action of the pulmonary vessels. The cardiac dilatation following the intravenous injection of arsphenamine does not appear to be a direct effect of the action of the drug but rather secondary to the pulmonary obstruction. The toxicity of various arsphenamine preps., as determined by their circulatory effects in the dog, varies considerably. The circulatory effects of an oxidation product, amino-hydroxyphenyl arsenoxide, were studied on dogs. Arsphenamine added to serum produces a ppt. *in vitro*. The ppt. between serum and the dihydrochloride is very bulky. The ppt. between serum and the mono-Na salt varies from a distinct turbidity to a moderately heavy ppt. No ppt. *in vitro* has been observed to occur between dog serum and alkaline arsphenamine solns. containing 0.8 cc. or more of N NaOH per 100 mg. of drug.

C. J. WEST

**Quantitative studies in chemotherapy. I. The trypanocidal action of antimony compounds.** CARL VOEGTLIN AND HOMER W. SMITH. U. S. Public Health Service. *J. Pharmacol.* 15, 453-73(1920).—The specific action of Sb lactate on the trypanosomes of infected rats can be studied fairly accurately by following the disappearance of the parasites from the blood stream. A sharply defined threshold (minimum effective dose) is observed, below which the drug has no appreciable effect upon the parasites. This threshold is in part due to the nature of the reaction between the drug and the parasite, and in part to absorption of the drug by the tissues of the host. The process curve is an orderly one and consistent throughout individual expts. However, various expts. indicate 2 general types of reactions, differing in reaction velocity. In 1 type the reaction velocity remains constant throughout the expt. In the other the reaction velocity is constantly increasing. **II. The trypanocidal action of arsenic compounds.** *Ibid* 475-93. Differences in the toxicity of various arsenic preps. have been explained on the hypothesis that they must be changed to one type, namely the trivalent oxide, RAs<sub>2</sub>O<sub>3</sub>, before exerting their principal action. Differences in the trypanocidal activity of these compounds have been explained upon the same hypothesis. **III. The oxidation of arsphenamine.** *Ibid* 16, 199-217(1920).—The Na salts of the following arsenicals are relatively very stable towards atmospheric O: Arsenious acid, Me and Et arsenious oxides, Ph and diphenylarsenious oxide, *p*-aminophenylarsenious oxide. Arsphenamine (di-HCl), contrary to the prevalent views, is exceedingly stable towards atmospheric O. The addition of alkali leads to a rapid increase in the rate of oxidation of the compound, the rate of oxidation being roughly inversely proportional to the OH ion concn. The Na salt of arsphenamine is 1st oxidized to the corresponding oxide, and this compound is simultaneously oxidized to the pentavalent arsenical. The relative concn. of unchanged arsphenamine and oxide depends on the rate of reaction. The slower the reaction the more oxide is formed, but in every case the last portion to be oxidized consists of 50% arsphenamine and 50% oxide. *m*-Amino-*p*-hydroxyphenylarsenious oxide ("arsenoxide") is oxidized only in alk. soln. The nature of the process curve indicates that the reaction is catalyzed by the reaction product. Nearsphenamine shows a rapid oxidation on exposure to air, amounting to about 50% in the 1st ten min., after which the rate decreases rapidly. The nature and rate of oxidation of arsphenamine and nearsphenamine to the corresponding oxides furnish an explanation of the increase in toxicity and trypanocidal activity of these compounds when their solutions are exposed to light.

C. J. WEST

The antagonistic actions of epinephrine and potassium chloride on the tonus and tonus waves in the excized terrapin auricles. CHARLES M. GRUBER. Univ. Colorado. *J. Pharmacol.* 15, 271-7(1920).—KCl causes an increase in the general tonus and tonus waves and may cause a disappearance of the contractions in the excized terrapin auricle. The average concn. for 55 expts. was 0.22%. The results with CaCl<sub>2</sub> were so variable that no deductions could be drawn. A synergistic action exists between KCl and CaCl<sub>2</sub> upon the general tone and tonus waves in the auricles. Epinephrine in proper doses causes a disappearance of the general tone and tonus waves aroused by KCl alone or in combination with CaCl<sub>2</sub>, causes a reappearance of the contractions if they have disappeared and increases the rate and the strength of contractions when still present. Cf. C. A. 14, 2220. C. J. WEST

Action of chloral on the pupil. E. G. HYATTI, HUGH MCGUIGAN AND F. A. RETTIG. Univ. Ill. *J. Pharmacol.* 15, 415-26(1920).—Large doses of chloral may produce a pin point pupil. The action is central and due to removal of inhibitory influences which are normally active. Strychnine, caffeine, atropine and other centrally acting drugs are antagonistic to the action of chloral. C. J. WEST

Perfusion of the medulla of the turtle. III. Epinephrine. A. D. BUSH. Univ. N. Dakota. *J. Pharmacol.* 15, 297-300(1920).—Epinephrine does not seem to exert, registerable influence on the cardio-inhibitory center of the striped turtle. Cf. C. A. 14, 1163, 2221. C. J. WEST

Mechanism of the toxic action of cyanogen chloride. C. I. REED. Univ. Kansas. *J. Pharmacol.* 15, 301-4(1920).—Death immediately following the administration of CNCl by any means is due to CN action entirely, though there may be other factors in cases of delayed death. Sodium thiosulfate will protect against the toxic action of CNCl, in the same way as against HCN. C. J. WEST

Tyramine as a morphine antagonist. HENRY G. BARBOUR AND LLOYD L. MAURER. Yale Univ. School of Med. *J. Pharmacol.* 15, 305-30(1920).—Tyramine (1.6 to 20 mg. per 100 g.) antagonizes the respiratory effects of morphine (1 to 1.8 mg. per 100 g.) when the two are injected in the same soln. The most complete antagonism was noted when 1 mg. morphine was injected with 6 to 20 mg. tyramine, the average max. decrease from normal volume amounting to only about 0.2. CO<sub>2</sub> (7-8%) breathing has practically no stimulating effect upon morphinized rats. It increases the stimulating effect of tyramine under normal conditions. After morphine CO<sub>2</sub> potentiates the tyramine antagonism where optimum doses of the latter are employed, but has a very feeble effect if the doses are smaller. Simultaneous injection of morphine and tyramine in separate subcutaneous regions results in a greater exhibition of respiratory depression than when the drugs are given in the same solution but this is followed promptly by an unusually marked antagonism. To account for all the results obtained it appears necessary to assume both a non-acid and an acid factor in the stimulating action of tyramine. C. J. WEST

Effect of emetine on *Entameba histolytica* in stools. WILLIAM ALLAN. Charlotte, N. C. *J. Pharmacol.* 16, 21-33(1920).—Using emetine and *Entameba histolytica* from various sources, in no instance did emetine affect the appearance or motility of these organisms in the stools in dilns. weaker than 1:2000 in the space of 1-2 hrs., and often dilns. as strong as 1:150 failed to kill the entamebae in the allotted time. C. J. WEST

The carminative action of volatile oils. J. W. C. GUNN. Univ. College. *J. Pharmacol.* 16, 39-47(1920).—The carminative action of volatile oils can best be explained by their relaxing and inhibiting the movements of plain muscle. Their other effects on the gastro-intestinal tract may be explained by their local irritant action, by reflexes arising therefrom, or possibly by acceleration of absorption. C. J. WEST

**Presence of histamine in extracts of the posterior lobe of the pituitary glands and on preliminary experiments with the pressor constituent.** JOHN J. ABEL AND T. NAGAYAMA. Johns Hopkins Univ. *J. Pharmacol.* 15, 347-99(1920).—Infundibular exts. that have been made with care from fresh glands, that is to say, with the avoidance of long boiling or of long exposure to acids, contain a small but readily detectable amt. of histamine. Exts. such as are employed in therapeutics contain larger quantities of this amine. Brief treatment of the freshly prepd. infundibular exts. with HCl on the water bath, or boiling such exts. under a reflux with 0.5% HCl for 0.5 hr., completely abolishes the blood pressure raising action of the ext. and causes a marked increase in the amt. of free histamine. Exts. which have been subjected to this treatment with acids always induce a pronounced fall in arterial pressure, in place of the rise of pressure usually observed with normal or untreated extracts. This fall is caused by 2 substances, B, a histamine like substance, which gives the Pauly reaction but not the biuret, is sol. in alc., but insol. in  $\text{CHCl}_3$ , and C, histamine. About  $\frac{1}{6}$  of the depressor effect on the blood pressure appears to be due to B and the remainder to C. The residual oxytocic activity is due to these same substances. These bodies are apparently not specific constituents of the infundibulum. Together they appear in animal exts. of all kinds and are easily obtained by hydrolyzing proteoses (see following abstr.). Preliminary observations in regard to the blood pressure raising constituent of the infundibulum are given. Highly active pressor salts are described, which are many times more powerful in their action on the uterus than histamine. Tetranitroaniline has been employed as a means of isolating the pressor and oxytocic constituent of the infundibulum. The action of HCl on these pressor and oxytocic derivs. appears to be quite like that observed when pituitary exts. are treated with acid. Tentative opinions are expressed as regards the chemical nature of the pressor substance.

C. J. WAST

**Histamine and a histamine-like substance as decomposition products of albumoses.** T. NAGAYAMA. Johns Hopkins Univ. *J. Pharmacol.* 15, 401-14(1920).—The proteoses of commerce vary in toxicity, as shown by comparative tests on the blood pressure and on the uterus. A secondary albumose prepd. from fresh thyroid glands by Abel and Rohde, by means of peptic digestion, was less active in its effects on the blood pressure and the uterus than the com. prepn. tested. Hydrolysis with HCl, under conditions described, causes the appearance of an appreciable quantity of histamine, and also of a histamine-like substance, similar, if not identical in character with that described above. Attention is called to the large amount of  $\text{CHCl}_3$ -sol. matter that is obtainable from the hydrolysis product of the proteoses studied. C. J. WAST

**Experimental investigations on the action of long-continued aniline inhalation.** RUDOLF JAFFÉ. Univ. Frankfurt, a. M. *Centr. allgem. Path. u. path. Anat.* 31, 57-63 (1920).—J. used 3 mice, 3 rats and 3 rabbits. The animals lived on an av. 6 months after expts. were begun on continuous inhalation of aniline, toluidine and naphthylamine vapors. One animal lived for 1 year and 4 months. In no case was there the development of a tumor in the urinary bladder. The lungs showed the most changes, high grade hyperemia, bronchitis and peribronchitis, finally broncho-pneumonia and abscess formation with necrosis. In a few cases fatty changes in the kidney epithelium and myocardium were found. In the liver, the aniline and toluidine animals showed fatty changes, the naphthylamine ones necrosis, regeneration, connective tissue increases and round cell infiltration. The findings in the liver, as also shown in exptl. cirrhosis, seem to indicate that aniline belongs to the poisons that injure the blood directly. This is shown by the iron pigment in the spleen and the masses of iron in the Kupfer cells of the liver. It would be interesting to see if true cirrhosis of the liver occurs often in workers with aniline or aniline compds. E. P. WOLF

The response of the animal organism to repeated injections of an active deposit of radium emanation. Intravenous injections in dogs. HALSBY J. BAGG. Memorial Hosp., N. Y. *J. Cancer Res.* 5, 301-19(1920); cf. C. A. 14, 1155.—B. found that large intravenous doses of active deposit of Ra Em produce a considerable reduction in the number of white blood cells up to 80% of their total number in an initial dose while the red blood cells show a simultaneous reduction of about 25%. Repeated doses amounting to 338.4 mc. distributed in 4 intravenous injections, produced a marked decrease in the number of circulating lymphocytes of the blood. Digestive disturbances following large doses were associated with a considerable reduction in body wt. A rise of body temp. followed treatment in several cases, probably an adaptive reaction to meet the toxic conditions produced by destruction of considerable amts. of cellular material. Metabolic changes showed a decided increase in the total N content of the urine as shown by urea, creatinine, uric acid and the phosphates. Relatively small doses given after a previous injection, even when metabolism was again back at normal, produced definite changes in the chem. content of the urine, indicative of destructive changes within the organism. On histological examn. the organs showed considerable congestion of the principal viscera. When the active deposit of Ra Em is used intravenously as a therapeutic agent, great care should be taken to grade the doses in accordance with the general physical condition of the patient, which should be detd. by frequent urine and blood analyses, and if more than one dose is given it should be smaller than the original dose. E. P. WOLF

Secalopan (UHLMANN, MIRMELSTEIN) 17.

#### I—ZÖÖLOGY

R. A. GORTNER

Axial gradients in Hydrozoa. III. Experiments on the gradient of *Tubularia*. LIBBIE H. HYMAN. Univ. Chicago. *Biol. Bull.* 38, 353-403(1920).—This exptl. work was undertaken as a reply to Banus (*J. Exp. Zool.*, 26, 265-75).—The existence of a metabolic gradient in the stem of *Tubularia* is shown by (a) differential susceptibility of apical and basal regions of the stem to  $\text{Et}_2\text{O}$  and to cyanide. Apical regions were more susceptible. (b) Differential capacity of the regions to reduce  $\text{KMnO}_4$ . The apical end has the greater reducing power. (c) Difference in electrical potential along the stem. Apical regions are negative to basal levels within the limits of the individual. (d) Difference in the rate of regeneration of apical and basal pieces. The paper reports work chiefly on this latter point. Apical regenerate on the whole more rapidly than basal. Rate of regeneration of pieces of *Tubularia*, depends, when other factors are equal, upon the level which those pieces occupied in the intact stem; it is more rapid the nearer the pieces lie to the original distal end of the stem. A metabolic gradient exists in the stem which is the primary cause of these regional differences in rate of regeneration. The exptl. results are assembled in 11 tables. L. W. RIGGS

Combined action of X-rays and of vital stains upon *Paramecia*. W. M. BALDWIN. Union Univ. *Biol. Bull.* 39, 59-66(1920).—The actions of Nilblau sulfate, Alizarinblau, S, Trypanblau, Isaminblau, Nilblau chlorhydrate, Trypanol, Dahlia neutral red, Aniline red (Sudan III, oil), Janus green and Methylene blue BX upon *Paramecia* were first studied, then the effect of X-rays acting alone and finally the action of X-ray energy while under the influence of these vital stains. The quantity of X-ray energy necessary to inhibit the activity of unstained *Paramecia* varies from 60 to 80 milliamperes min, under the conditions of these expts. When cultures are stained the amt. of energy required to inhibit their activity is reduced to from 5 to 10 milliamperes min. The cause of death appears to be due to "an assumption of stain by the nucleus." The expts. suggest that the cause for the increased susceptibility of the organisms to X-ray energy

is to be found in some factor which is operative only when X-rays act on cells in the presence of the stain.

L. W. RIGGS

## 12—FOODS

W. D. BIGELOW AND F. F. FITZGERALD

**Absorption of cyanide gas by foodstuffs.** ANON. *Public Health Reports* 35, 1597(1920).—The question of the possibility of adsorption or absorption and retention of CN gas by foodstuffs exposed in the process of fumigation of vessels, warehouses, and similar places, to the extent that they are rendered dangerous or injurious to consumers has arisen from time to time. The matter has recently been carefully investigated by the U. S. P. H. S. by the practical method of exposing foodstuffs to the fumes of CN gas and then feeding them to animals. The conclusion from these expts. is that the possibility of food poisoning occurring from food materials exposed to CN gas is extremely remote.

JULIAN H. LEWIS

**New methods of food dehydration.** K. GEORGE FALK. *Am. Food J.* 15, No. 2, 15-7(1920).—An address (cf. *C. A.* 14, 82).

H. A. LEPPER

**Some scientific aspects of cold storage.** JOSEPH SAMUEL HEPBURN. Hahnemann Med. Coll., Phila. *Hahnemannian Monthly* 55, 708-13(1920).—Résumé of the biochem. changes occurring in cold-stored *poultry, eggs and fish*. JOSEPH S. HEPBURN

**Heat penetration in processing canned foods.** W. D. BIGELOW, G. S. BOHART, A. C. RICHARDSON AND C. O. BALL. *Bull. Res. Lab. Nat. Cannery Assoc.* No. 16-L, 128 pp.(1920).—The development of heat-penetration studies is discussed and an app. and a method for measuring heat penetration together with an experimental rotating cooker are described. The laws of conduction and convection govern the penetration of heat from the outside to the center of the can. The max. heat penetration of canned food is that of H<sub>2</sub>O or slightly less and the min. may be regarded as that of a body consisting largely of H<sub>2</sub>O, but in which the H<sub>2</sub>O is distributed in minute cells that entirely prevent convection. The influence of the size of can, initial and retort temp., consistency of product, processing in dry steam or under H<sub>2</sub>O, lack of uniformity of product, and rotation on heat penetration are discussed. Asparagus, beans (string and baked), beets, cherries, coconut, corn, elderberries, gooseberries, hominy, meat, milk, olives, peas, plums, salmon, spinach, squash, pumpkin, sweet potatoes, and tomatoes were studied. Heat penetration curves are calcd. and discussed. The original must be consulted for full discussion and conclusions on above mentioned foods.

H. A. LEPPER

**Preservation of tomatoes in tins.** P. CARLES. *L'industrie française de la conserve* 5, No. 25, 391-3(1918); *Bull. Agr. Intelligence* 10, 621.—The acidity of tomato pulp has little, if any, effect upon the tin if the can of fruit is of good quality and ripe when concd. to 15 g. dry matter per 100 (French standard). Presence of salt causes action on tin by liberation of HCl due to mass action of vegetable acidity. When pulp is late season or not 1st quality the acidity is greater, owing to lack of sugars, pectin substances or other "metal isolators" and has more action on the metal. H. A. LEPPER

**The process of ripening in the tomato considered especially from the commercial standpoint.** CHARLES E. SANDO. Bur. Plant Industry, U. S. Dept. Agr., *Bull.* 859, 1-38(1920).—With the object of discovering the chem. basis for the inferiority of commercially picked and ripened Florida tomatoes marketed in the North during the winter and spring, a series of analyses was made of tomatoes of several degrees of maturity and of tomatoes ripened artificially under various conditions of ventilation. The results obtained indicate that in general throughout the ripening period there is an increase in moisture, acids and sugars, and a decrease in solids, total N, starch,



pentosans, crude fiber and ash. Carbohydrates undergo the most striking change during ripening. Sugars increase from 25.66% in fruit 14 days old to 48.32% in ripe fruit. Starch decreases in the same interval 15.84 to 2.65%. The percentage compn. of fruit picked green but ripened with free access of air compared with analyses of turning and vine-ripened fruit did not show enough variation to account for the great differences in taste found in commercially shipped fruit. Lack of ventilation was found to have a marked effect on the compn. and quality of the fruit, and it is concluded that wrapping probably modifies the course of ripening to such an extent as to account for marked changes in taste and flavor, and that the combined results of picking fruit green, of wrapping and of closing the cars in transit probably account for the total differences existing in quality between commercially shipped and vine-ripened tomatoes.

W. H. ROSS

Work and objects of the institute for research in potato cultivation. KARL SNELL. Berlin-Steglitz. *Z. Spiritusind.* 43, 313(1920).—A review of the work of the institute on pure seed, fertilizer values, insect enemies and diseases, storage and allied problems.

W. B. V.

Comparative value of quantitative and qualitative bacteriological methods as applied to milk with special consideration of the methylene blue reduction test. E. G. HASTINGS. *J. Dairy Sci.* 2, 293-311(1919).—The four methods for the detn. of the no. of bacteria in milk which were studied are the plate culture method recommended by Am. Public Health Assoc., the Breed method or direct microscopic count, the Frost "little plate" and the methylene blue reduction method. Original data show that the methylene blue reduction test measures the bacterial content of milk with a high degree of accuracy when applied to samples the bacterial content of which has been varied by the addition of different amts. of the same inoculum. It may well replace the ordinary plate culture or direct count methods and it measures the bacterial content as accurately as any method now available.

H. A. LEPPER

An unusual outbreak of ropy milk. B. W. HAMMER AND W. A. CORDES. *J. Dairy Sci.* 3, 291-9(1920).—An outbreak of ropy milk occurring in Jan. 1920, an unusual time of year, was found to be due to a new microorganism. It was given the name *Staph. cremoris-viscosi* and was found to be closely related to *M. mucofaciens* of Thöni and Thaysen and somewhat like *M. freudenreichii* of Guillebeau. Morphology, culture characteristics and biochem. features are given.

H. A. LEPPER

Report of committee on milk supply of the Sanitary Engineering Section, American Public Health Association, August, 1920.—This rept. (published in pamphlet form) contains up-to-date information on milk pasteurization. The report covers the present status of milk pasteurization, the effect of pasteurization on the compn. of milk, the process of milk pasteurization, the analytical control of pasteurization plants, and state and municipal supervision.

H. A. WHITTAKER

Effect of temperature on the fat column in the Hortvet test bottle. J. HORTVET. *Dairy Rec.* 21, No. 21, 26-8(1920).—Using the values obtained with the Mojonner tester as a standard of reference it was found that a temp. of 125-130 F. yielded the best results.

H. F. ZOLLER

The variation of butter-fat percentage with age in Jersey cattle. JOHN W. GOWEN. Maine Agr. Expt. Sta. *Bull.* 290, 133-44(1920).—The mean for the herd was  $5.2260 \pm 0.0073$  which was as high as that for any of 28 different breeds (ranging from 3.05 to 5.12). The frequency distribution for this range is bi-modal, one mode occurring at about 3.7% and the other at about 5.0%. The variation from cow to cow is about twice as much as that of the solids-not-fat %. There is a slight negative correlation ( $-0.1126 \pm 0.0161$ ) between the age of the cow and the butter-fat % which the cow will produce.

ALBERT R. MERZ

**Meat-vegetable preserves.** A. KOSSOWICZ. *Z. Fleisch-Milchhyg.* 27, 49-52 (1920).—The preservation of meat-vegetable mixts. such as gulasch, etc., are studied from the standpoint of sterilization temps. and pressures necessary to destroy *B. putrificus* and other gas-forming organisms. H. F. Z.

**Utilization of kid, rabbit, horse and seal meats as food.** ARTHUR D. HOLMES AND HARRY J. DEUEL, JR. *J. Ind. Eng. Chem.* 12, 975-9(1920).—The meats of kid, rabbit, horse and seal are discussed and analyses given. The digestibility of the protein of these four kinds was essentially the same as that of other and better-known meats.

H. A. LEPPER

**The use of mussels as a food for poultry in Denmark.** N. M. T. STENBOECK. *Ugeskrift for Landmaend* 64, 178(1919); *Bull. Agr. Intelligence* 10, 719-20.—Mussels, boiled, washed, dried and reduced to a meal ("mushingemel") fed to poultry gave good relative egg production. The meal which will keep a long time contains,  $H_2O$ , 3-4; protein, 8-10; fat, 1-2; other org. matter, 13-14; mineral constituents (especially  $CaCO_3$ ) about 75%.

H. A. LEPPER

**"Babasso" nuts and their oil.** A. DIETRICH AND L. KNÖRR. *Neuss. Z. Nahr. Genussm* 40, 162-3(1920).—A description of the Brazil nut (*Attalea funifera*) which yields an oil similar to coconut oil. Analyses of the seeds and consts. of the oil are given.

L. D. ELLIOTT

**The hygroscopic moisture of flour exposed to atmospheres of different relative humidity.** C. H. BAILEY. *J. Ind. Eng. Chem.* 12, 1102-4(1920).—The moisture content of flour in equil. with the atm. is a function of atm. humidity. The rate at which equil. in moisture content is approached apparently depends upon conditions of exposure. Hygroscopic moisture in flour which was in moisture balance with atm. humidity at 25° ranges from about 5 1/4% of moisture at 30% relative humidity to 15% of moisture at 80% relative humidity.

C. H. BAILEY

**Dilution of bread with rolled oats.** PAROW. *Z. Spiritusind.* 43, 303(1920).—This is an argument in favor of oats for use in bread, as against rolled oats only, based principally on the fact that if the available quantity of that substitute were diminished, potatoes would be partly withdrawn from their other uses to make up the deficiency.

W. B. V.

**Serological detection of corn-cockle in meal and bran.** J. BECKER. *Centr. Bakt. Parasitenk., Abt. II* 48, 417-20(1918).—The presence of corn-cockle in food can most certainly and simply be detected by means of the precipitin reaction, especially if the samples are small and contain a small amt. of corn cockle.

JULIAN H. LEWIS

**Italian baking powder.** KURT BRAUER. *Chem.-Ztg.* 44, 642(1920).—The compn. of one sample of Italian baking powder is given and discussed.

H. A. LEPPER

**The functions of lye in olive processing.** W. V. CRUESS. *Fig & Olive J.* 4, No. 2, 4-6(1919); *Exptl. Sta. Rec.* 42, 113-4.—Lye (0.3-0.5%) generally added to standing solns. which is considered to hold harmful bacteria in check and improve pickling qualities, actually favors growth of bacteria, salt preventing bacterial action. If lye in 1st treatment wherein olives are darkened is too concd. the color will be dissolved out, particularly if the exposure treatment is used instead of running or aerated  $H_2O$ . The removal of bitterness by the last lye treatment is believed due to the destruction of glucose and not to a neutralization. Excess of lye necessary beyond neutn. is 0.25% (8 lbs. lye per ton of olives).

H. A. LEPPER

**Gluten in Italian pastes.** A. CUTOLO. *Bol. soc. nat. Napoli* [2] 10, 130-69 (1917); *Exptl. Sta. Rec.* 42, 162.—An outline of process of manuf., analyses of various types of Italian food pastes and exptl. studies on changes taking place in the gluten of the flour in the manufacture of the pastes are given. The agglutinating property of the gluten is lowered and not its solubility by enzyme action, especially during drying. The amount of extractable gluten is thought to be an index of the quality of the paste

and the nearer it approaches the total N the better the quality from organoleptic, com. and gastronomic points of view.

H. A. LEPPER

Some aspects of chemical research in the flavoring extract industry. MELVIN DE GROOTE. *Am. Perfumer* 15, 253-6, 290-2, 300(1920).

E. H.

A comparative study of corn silage in concrete and stave silos. R. H. SHAW AND R. P. NORTON. *J. Dairy Sci.* 3, 300-7(1920).—Rather complete chem. analyses including volatile acids of corn before ensilage and of 3 samples held in small sacks at different points in the 2 silos after ensilage revealed no marked differences due to the type of silo. The temp. during ensilage next to wall, 18 in. from wall, and at center showed no marked differences due to type of silo. Quality from each was judged to be the same, as cows ate both with avidity.

H. A. LEPPER

Cattle feeding with the by-products of the Erytrean corozo nut. Experiments in Italy. BELTRAMI. *L'Italia agricola* 56, 51-3(1919); *Bull. Agr. Intelligence* 10, 588.

—The by-product from the manuf. of buttons from the Erytrean corozo nut (*Hyphaene thebaica*) is a white, light, odorless meal. Feeding expts. showed it to be of good food value and digestibility. Its analysis (1st figure) compared with American corozo (2nd figure) is H<sub>2</sub>O, 10.35-11.05; ash, —, 1.10; crude protein, 4.70-4.31; fat, 10.20-1.03; cellulose, 14.00-4.20; pentosans, 2.10-2.20; N-free ext. (except pentosans) 59.00-76.11%.

H. A. LEPPER

Fodder poisoning and its causes. G. B. VAN KAMPEN. *Olien en Vetten* 4, 871-3, 643-6; 5, 1-3.—A summary with references.

NATHAN VAN PATTEN

The use of the contents of the rumen as cattle food. I. Researches in Italy.

E. ARUCH AND BELTRAMI. *L'Italia agricola* 55, 236-40(1918); *Bull. Agr. Intelligence* 10, 586-7.—The contents of the rumen can be made into utilizable food, being best and most cheaply prepared by drying in the sun followed by desiccation in a stove. Rapid washing followed by drying in thin layers will remove the disagreeable odor. The contents of the rumen washed and dried gives H<sub>2</sub>O 7.86, ash 2.50, N matter 6.25, fat 1.70%, a compn. very similar to that of average hay. Feeding expts. are given.

H. A. LEPPER

BEYTHIEN, A., HARTWICH, C. AND KLIMMER, M.: *Handbuch der Nahrungsmitteluntersuchung*. Bd. III. Bakteriologischer und biologischer Teil. Edited by M. Klimmer. Leipzig: Verlag von Chr. Herm. Tauchnitz. 648 pp. M. 55.

Bread. R. L. CORBY. U. S. 1,355,127, Oct. 12. Proper conditioning of the dough mass in making bread is attained with relatively small amt. of yeast by the addition to the flour used in making the dough of a mixt. of H<sub>2</sub>O 82 lbs., sucrose 3.5 lbs., and NaCl 2 lbs. for every 100 lbs. of flour and 2.5 lbs. of yeast; or, preferably, by replacing the sucrose by a material rich in maltose, dextrin and dextrose and using a much smaller proportion of yeast. U. S. 1,355,128 relates to a method of producing a yeast-economizing and dough-conditioning compn. of the latter class by forming a liquid mass containing saccharine substances of the maltose class and proteins, acidifying the mass to the point where the diastatic and proteolytic materials are rendered inert, then more highly acidifying the mass. The acidification is carried to the point where the acid will be sufficient properly to condition the gluten of the dough flour, but not to prevent nourishment of yeast by the proteins and saccharine substances of the compn.

Bread-making ingredient. R. L. CORBY. U. S. 1,355,129, Oct. 12. A compn. for use with flour in prepg. a bread dough batch is prepd. as follows. Rye malt, barley malt or barley sprouts or similar material is heated with H<sub>2</sub>O to 50-75° for 2-5 hrs. to effect liquefaction of the starch and production of maltose, dextrose and dextrins. Sucrose is added and the mixt. is subjected to the action of a culture of lactic acid

bacteria which act upon the maltose, dextrose and dextrans with production of lactic acid and render the diastatic and proteolytic enzymes substantially inert, and further act upon the sucrose to convert it into invert sugars, with further production of a material proportion of free acid. After this acidification, the material is heated to 85–95° which coagulates substances such as undigested proteins which might discolor dough in which the compn. is used. Insol. substances are filtered out and the filtrate is concd. by evapn. and sterilized.

**Milk-fat from milk and cream.** E. B. PHELPS, A. E. STEVENSON and J. C. BAKER. U. S. 1,354,883, Oct. 5. In obtaining pure milk fat the cream is sepd. from skim milk and the remaining milk serum is dild. out of the cream by washing with H<sub>2</sub>O to render the emulsion unstable and the fat particles capable of substantially complete coalescence and ready sepn.

**Desiccating buttermilk.** N. P. COLLIS. U. S. 1,356,340, Oct. 19. Buttermilk containing curd particles in suspension is agitated to prevent pptn. of the curd and the material is sprayed on a heated surface to effect evapn.

**Pasteurizing apparatus.** O. EICK. U. S. 1,356,055, Oct. 19.

### 13—GENERAL INDUSTRIAL CHEMISTRY AND CHEMICAL ENGINEERING

HARLAN S. MINER

**Chemical industry and trade of Sweden.** O. P. HOPKINS. *J. Ind. Eng. Chem.* 12, 1045–54(1920). E. J. C.

**The chemical industry (of Germany), retrospect and outlook.** HANS GOLDSCHMIDT. *Z. angew. Chem.* 33, I, 257–60(1920).—An address. E. J. C.

**Report of the activities of the German Board of Trade during the war.** ANON. *Chem. Met. Eng.* 23, 976–9(1920).—A summary is given of the report on conditions in the chem. and allied industries, with tabulated data on the wage conditions and accidents for the period 1913–18. E. H.

**Grasselli Medal award (to Allen Rogers).** *J. Ind. Eng. Chem.* 12, 1127–8(1920); *Chem. Met. Eng.* 23, 794(1920).—The medal was awarded to Dr. Rogers for his paper on "Industrial uses for the shark and porpoise" (cf. *C. A.* 14, 1062). E. J. C.

**The (British) factory inspector's report for 1919.** ANON. *Chem. Trade J.* 67, 467–9(1920).—An abstract of the report which deals with accident prevention, explosions, and industrial diseases with comparisons of the numbers of cases with those of former years. All Cu and brass fittings were removed from vessels used in the manuf. of synthetic AcOH. W. H. BOYNTON

**Industrial readjustments of certain mineral industries affected by the war.** GUY C. RIDDELL, et al. U. S. Tariff Commission, *Tariff Information Series* 21, 320 (1920).—A compilation of the tariff information surveys dealing with the industries producing Sb, chromite, graphite, magnesite, Mn, potash, pyrites, S, Hg, and W. These important minerals and metals were used during the war, either directly or indirectly, for military purposes and readjustment to peace conditions has been slow and difficult. Detailed information is given concerning sources of supply, production, imports, market prices, competitive conditions, and so far as possible, foreign and domestic costs of production. S. D. KIRKPATRICK

**Nature and use of lubricants.** K. A. WENIGER. *Papierfabr.* 18, 704–6(1920).—A general discussion of animal, vegetable, and mineral lubricants, with special reference to lubrication in the paper mill. C. J. WEST

**Electric insulation materials.** *Kunststoffe* 10, 161–4, 171–3(1920).—A list of patents. C. J. WEST

Tanks and pipe lines as causes of accidents. HOMER A. HOFFMAN. *Chem. Met. Eng.* 23, 1023-5(1920).—A study has been made of the hazards involved in the use of storage tanks and pipe-line distributing systems in chem. plants and safe methods are suggested for the construction, cleaning and repairing of underground and overhead tanks and pipe lines. E. J. C.

Flow of oil in pipes. I. ARTHUR C. PRUSTON. *Chem. Met. Eng.* 23, 607-13 (1920).—Hydraulic theory has been developed for water, but its laws, as usually stated, do not apply to liquids with different kinematic viscosity, where  $\eta$  is the viscosity in absolute units,  $i. e.$ , poises, and  $\rho$  the density. P. attempts to det. the friction loss of any liquid as a function of its viscosity, based on work of the Nat. Phys. Lab. and Bur. of Standards. Reynolds has shown that the motion in a pipe is linear until  $v\rho d/\eta = R$  is equal to 2050, where  $\rho$  = the density,  $v$  = velocity, and  $d$  = diam.; the motion is turbulent when  $R = 2900$ . For intermediate values of the criterion  $R$ , there is a mixed régime. For each of the three régimes the relation between the friction head  $h$  and the velocity is expressed by the equation  $h = Kv^m$  where  $K$  is a const. and  $m$  is unity in the viscous régime, 1.74 in the turbulent regime and about 3.0 for the mixed régime, the exact value in the last case being indeterminate. Using Reynold's criterion (1) the range of velocities in the viscous régime and the loss of head may be calcd. by the formula  $h = Kv$ , (2) the range of velocities and loss of head in the turbulent régime is calcd. by use of the formula  $h = Kv^{1.74}$  (3) and in the mixed régime by  $h = Kv^3$ . Work in the Nat. Phys. Lab. shows that for equal values of  $R$  the friction factor is always the same and may be calcd. by the Fanning formula  $h = f v^3/2 dg$ . P. shows the relation between  $R$  and  $f$  graphically. II. *Ibid* 685-9.—P. gives tables for the friction head in ft. per hundred ft. of pipe for liquids of different viscosities. He also gives corrective factors for pipe other than smooth drawn steel or brass pipe. EUGENE C. BINGHAM

Indication of a mechanical theory of fractional distillation. E. CHENARD. *Industrie chimique* 7, 146-9(1920).—The elementary bases of technical distn. and their divergencies are discussed. In an attempt to explain these divergencies, C. states that the state of vapor is but poorly defined and advances the hypotheses of the co-existence of liquid and gaseous states in the vapors, and that a vapor is a mixt. of a liquid in suspension and a perfect gas. This is against the accepted theory which is, however, based upon observation. C. gives as the 2 effects of compression, the condensation of a part of the gas to liquid and the compression of a perfect gas. The conclusion is that equal masses of  $H_2O$  vapor changing under the same conditions of temp. and pressure without exterior heat influence (adiabatically), behave differently, depending upon the conditions of temp. and pressure under which they were at first vaporized. P. D. V. MANNING

"Autovapor" electrical evaporating system. F. WIRTH. *Bull. Schweiz. Elektrot. Verein* 10, 347-55; *Science Abstracts* 23B, 142-3.—This process for the evapn. of solns. offers a means of utilizing hydroelec. energy instead of coal and is more efficient than the direct use of elec. heating. The soln. to be evapd. is brought to its b. p. by means of a steam coil. The vapor formed by the boiling is drawn off, compressed and delivered into a coil in the evapg. vessel. Here it is condensed and causes the evapn. of more of the soln. The hot condensate is then run into a pre-heater which contains a coil through which the soln. is circulated before it enters the evaporator. From this pre-heater the cooled condensate is removed as distd.  $H_2O$ . In a plant which used this system for the evapn. of soda lye from 5% to 17%, the time required was 6 hrs. During 1 hr. in the middle of the 6 the following data were collected: 1070 kg.  $H_2O$  were removed from the lye by the compressor, 54.25 kw. hrs. were required for driving the motors, etc., the pressure in the evaporator was 1.29 kg. per sq. cm., the temp. of the lye 107.6°, temp. of the condensate leaving the feed heater 91.5°, 100 kg. of live

steam were supplied, the  $H_2O$  extd. per kw. hr. was 19.6 kg. excluding the live steam or 18 kg. allowing for the latter.

G. W. STRATTON

**Separation of slimes from liquids by centrifuging.** BERTHOLD BLOCK. Charlottenburg. *Chem. App.* 7, 139-40, 146-50(1920).—Second supplement to B.'s article, *C. A.* 14, 1872, 3505. Other supplements will follow.

J. H. MOORE

**Gas warfare (history of the defence).** J. W. TURNER. *Cairo Sci. J.* 9, 135-41 (1917).

E. H.

Scientific literature and industrial research (DUNSHEATH) 2. *p*-Cymene as a solvent (WHEELER) 2. Influence of electrolytic dissociation on the distillation in steam of the volatile fatty acids. Applications of the method of distillation in steam (REILLY, HICKINBOTTOM) 2. Mixing solids with liquids (Brit. pat. 148,124) 24.

**Chemical Engineering Catalog, 1920.** New York: The Chemical Catalog Co., Inc. 1450 pp. \$7.50. Cf. *C. A.* 14, 315.

VATER, R.: *Technische Wärmelehre (Thermodynamik)*. 2nd Ed. Edited by Fritz Schmidt. Leipzig and Berlin: B. G. Teubner. 122 pp. M. 5.60.

**Separating gases by liquefaction.** GRS. FÜR LINDE'S EISMASCHINEN AKT.-GES. Brit. 147,956, July 11, 1917. Addition to 24,735, 1914 (*C. A.* 10, 1699). The supply of heat to the base of the rectifying column and the withdrawal of heat from the top of the column are so arranged that the heat exchange within the column is at least equal to the heat of vaporization of the whole mass under treatment in the column.

**Cleaning gases.** FRANK R. MCGEE. Can. 206,156, Nov. 30, 1920. A flowing stream of impure gases is divided into smaller annular streams to which a whirling motion is given to maintain the impurities in an annular layer by centrifugal force while the gases are expanded centripetally to form a core of cleaned gases, which is delivered into a gas-collecting chamber, and the impurities are diverted into a chamber in which a flow of gases is induced to prevent back pressure. App. is also specified.

**Separating mixed gases by liquefaction and rectification.** F. E. NORTON. U. S. 1,354,056-7-8-9, Sept. 28. The pats. relate to app. and methods of manipulation especially adapted for treating air or water gas.

**Refrigerating gas formed from sulfur dioxide and propane.** A. G. CRAWFORD. U. S. reissue 14,955, Oct. 12. See original pat. 1,325,666, *C. A.* 14, 455.

**Recovering volatile solvents from air.** U. J. LEBOURVEAU and A. M. TAYLOR. U. S. 1,355,401, Oct. 12. Solvent vapors such as ether,  $C_6H_6$ , PhMe, alc. or EtOAc, which are mixed with air, are absorbed in acetone oil and then recovered from the oil by distn. U. S. 1,355,402 relates to the use of aniline instead of acetone oil for the same purpose.

**Method of heating materials during destructive distillation.** F. RIPP. U. S. 1,355,268, Oct. 12.

**Plastic metallic packing.** B. W. GOODSSELL. U. S. 1,355,830, Oct. 19. A packing material suitable for use in stuffing boxes is formed of Babbitt metal, Pb wool, mica flour, paraffin or beeswax and lubricating oil. The metals constitute about 92% of the mixt.

**Comminuting an alloy of white metal and lead.** W. LEWICKI. U. S. 1,355,984, Oct. 19. A material suitable for use in plastic metallic packings is formed by allowing an alloy of white metal and Pb in molten condition to flow in thin jets from a melting vessel, forming the jets into thin elastic leaves or scales by a jet of compressed air or other gas and projecting the product against a corrugated wall, from which it falls.

**Lining for tanks.** F. SCHULER. U. S. 1,355,360, Oct. 12; Ger. 318,033, Sept. 14, 1918. A lining for tanks which may be used to hold corrosive liquids is formed of a thin layer of stoneware with a metal insertion in it directly applied to the walls of the tank, and alternate thin linings of silicates such as glass tiles and layers of stoneware on the base layer.

**Filling materials for reaction towers, etc.** S. SAKAI. Japan 35,834, Feb. 14, 1920. Porcelain in special forms is specified.

#### 14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

**Relationship of the hydrogen-ion concentration of natural waters to carbon-dioxide content.** R. E. GREENFIELD AND G. C. BAKER. *J. Ind. Eng. Chem.* 12, 989-91(1920).—Equations are developed for calcg. the H-ion concn. of natural waters in which the CO<sub>2</sub> and bicarbonate content are expressed as ordinarily detd. These equations are less accurate with low bicarbonate concns. and do not apply to waters alk. to phenolphthalein.

G. C. BAKER

**The fluorine content of mineral waters.** P. CARLES. *Rev. chim. applicada* 4, 181-91(1919).—The analyses of mineral waters from 93 representative European localities show that 88 contain more than 0.001 g. NaF per l. Alk. waters have the highest content while those high in SiO<sub>2</sub> have the lowest. The av. is 0.007 g. per l.

C. B. SLAWSON

**The fluorine in the mineral waters of Portugal and Spain.** A. J. FERREIRA DA SILVA AND ALBERTO D'AGUILAR. *Rev. chim. applicada* 4, 202-6(1919).—The analyses of 3 mineral waters show an av. of 0.0235 g. of NaF per l., which is over 3 times the av. content of other European waters (cf. preceding abstract.)

C. B. SLAWSON

**Novel application of copper sulfate to basin walls for control of algae.** GEORGE F. GILKISON. *Iowa Sect. Am. Water Works Assoc.* 1920; *Eng. Contr.* 54, 468-9(1920).—Draining down the basins and spraying on the walls a soln. of 6 lbs. CuSO<sub>4</sub> to 10 gal. H<sub>2</sub>O, followed after 1 hr. by scrubbing with wire brush has been found most effective. 1 gal. soln. will treat 125 sq. ft. of wall surface, taking 2 men 4 min. This keeps the walls free from algae. Also in *Can. Eng.* 39, 596 (1920).

LANGDON PEARSE

**Features of new water works at Minot, North Dakota.** F. BASS AND E. J. THOMAS. *Munic. County Eng.* 59, 142-4(1920).

LANGDON PEARSE

**Innocence or repentance in drinking water.** H. W. CLARK. *N. E. Water Works Assoc.* 1920; *Can. Eng.* 39, 478-9(1920).—Twenty-five years ago a pure water was preferred to an impure water purified according to Dr. Drown. The records in Mass. show practically the same typhoid fever death rates with ground water and treated surface water supplies. Filtration followed by Cl is most effective.

LANGDON PEARSE

**Removing iron from public water supplies at Shelby, Ohio.** PHILIP BURGESS. *Central States Sec. Am. Water Works Assoc.* 1920; *Munic. County Eng.* 59, 175-8(1920).—The water contains Fe from 5 to 16 p. p. m. Old devices for removal included aeration and pressure filters. The new plant (1 million gal. per 24 hr.) has an aerator tower, 1 hour sedimentation, 6 hour clear well, and 2 filters (area 0.011 acre). The aerator consists of 3 trays, 12 in. deep, 12 inches apart, filled with coke. The plant reduces Fe to 0.3 p. p. m.

LANGDON PEARSE

**Filter-plant operation at Harrisburg, Pa. water department.** *Water Dept. Ann. Rep.* 1920; *Eng. Contr.* 54, 369(1920).—Filtered water in 1919 cost \$9.34 per mil. gal. The av. daily use was 107 gal. per capita, 85.7% of services are metered. The use of alum averaged 0.588 grains per gal. Soda ash was required 29 days. The av. period of filter service was 16 hr. 49 min.

LANGDON PEARSE

Experiments of the metropolitan water board (London) with rapid filtration. A. C. HOUSTON. 13th Research Report, *Engineer*, Sept. 3, 1920; *Eng. Contr.* 54, 309-11 (1920).—Studies made on stored Thames R. water showed that (1) sterilization is necessary with rapid filtration; (2) rapid filtration, even without a coagulant, will remove all coarse material and a large proportion of the fine; (3) color would be noticeable 3 mos. of the year; (4) rapid-filtered water would contain some suspended matter if no coagulant is used; (5) plankton are largely removed, preventing after-growth in pipes. The object of the studies was to save cost of coagulant. LANGDON PEARSE

Purification of swimming-pool waters. A. S. WOORTON. *Pacific Coast Park Boards* 1920; *Can. Eng.* 39, 493(1920).—Continuous purification is desirable. Coarse solids can be removed by a strainer, fine solids by a filter. Org. matters can be destroyed by filtration and aeration. Bacterial life can be destroyed by filtration and sterilization by chemicals, ozone or ultra-violet rays. LANGDON PEARSE

Experiences of the Montreal Water and Power Co. in the manufacture of filter alum. JAMES O. MEADOWS. *J. Am. Water Works Assoc.* 7, 841-4(1920).—See C. A. 14, 2831. E. H.

Fitchburg sewage-treatment plant. HARTWELL AND ALLEN. *Ann. Report Dept. Pub. Works* 1919; *Pub. Works* 49, 399-403(1920); *Eng. Contr.* 54, 458(1920).—This plant has been operated carefully for 6 yrs. During 1919, 98% of the settleable solids were removed by the Imhoff tank. The sprinkling filter received 2.69 mil. gal. per acre per 24 hrs., producing a non-putrescible effluent on a filter 10 ft. deep. The secondary tanks produced 93.28 tons of dry solids as compared with 823.7 tons in the Imhoff tank. The cost of operation was \$11,596.07. The removal of large amts. of grit from the Imhoff tanks and repairs made are described. LANGDON PEARSE

Experience with Imhoff tanks in Minnesota. J. A. CHILDS. *Munic. County Eng.* 59, 162(1920).—Since 1911, 40 municipal and institutional tanks have been installed. Experience has shown need of at least 2 cu. ft. per capita sludge capacity. An institutional tank through neglect was bothered by excessive scum, sometimes 11 ft. thick. Intelligent care in operation is essential. The Imhoff tank has been thoroughly satisfactory. LANGDON PEARSE

Garbage incinerator at White Plains. *Pub. Works* 49, 337-8(1920).—Garbage, ashes and rubbish are burned in a 20-ton incinerator costing \$12,000. L. PEARSE

Columbus municipal reduction plant. ANON. *Am. Soc. Mun. Impvt.* 1920; *Eng. Contr.* 54, 458(1920); *Pub. Works* 49, 441-2(1920).—All tannage is dried first to 10-12% moisture, degreased with gasoline, screened through 1/2-inch mesh, mixed with stick to run and then dried to 3%. Mixing is important. Av. (annual) analysis of tannage in percent is NH<sub>3</sub> 3.75; potash 1; bone phosphate 7.5. The grade of gasoline specified will distil off 95% or more at 300° F. Loss of solvent is 1 gal. per ton of garbage. In 1915, 218 lbs. of garbage were collected *per capita*, in 1918, 138; 1919, 157. LANGDON PEARSE

Sewage disposal at a Saskatchewan Penitentiary. R. F. UNICKER. *Eng. Contr.* 54, 405(1920). LANGDON PEARSE

Septic tanks for unsewered districts. C. E. KEEFER. *Pub. Works* 49, 388(1920).—For districts not connected to the trunk sewers, septic tanks have been built based on a minimum flow of 80 gal. *per capita* per 24 hrs., and a detention period of 8 hrs., with a foot of sludge in the shallow end. The sludge is pumped into tank wagons for disposal. LANGDON PEARSE

The Chicago drainage canal, today and tomorrow. F. C. SHENEHON. *Munic. County Eng.* 59, 154-5(1920).—This is a brief review of the status of the canal and the litigation of the Sanitary District, outlining a possible settlement covering control of the lake levels, treatment of sewage and diversion of 10,000 cu. ft. per sec. from Lake Michigan. LANGDON PEARSE



The Miles acid process on tannery waste. E. S. DORR. *Am. Soc. Mun. Impel.* 1920; *Pub. Works* 49, 403-4(1920).—A sample of tannery waste (containing solids 6449 p. p. m.) was treated with  $\text{SO}_2$  at a rate of 860 p. p. m. Sludge was pptd. at a rate of 4 tons (dry) per mil. gal. containing 16.88% grease and 7.5%  $\text{NH}_3$ . The effluent was inodorous and sterile, and contained only 50% of the original solids.

LANGDON PEARSE

BACH, H.: Das Kokereinebenproduktenabwasser im Emschergebiet und Versuche zu einer Reinigung. Essen: Emschergenossenschaft Essen. 58 pp. For review see *Gesundh. Ing.* 43, 524(1920).

BRINKHAUS, PAUL: Anlagen zur Gewinnung von natürlichem und künstlichem Grundwasser, Die Vorarbeiten, der Entwurf und Bau. Oldenbourg technische Handbibliothek. 23 Bd. Munich: R. Oldenbourg. 227 pp. M. 20.

KÖRTING, JOH.: Heizung und Lüftung. 3rd Ed. Berlin and Leipzig: Vereinigung wissenschaftlicher Verleger. Bd. I. M. 1.80. Bd. II, M. 2.10. For review see *Gesundh. Ing.* 43, 502(1920).

Softening water with base-exchanging silicates. T. R. DUGGAN. U. S. 1,354,604, Oct. 5. Water-softening devices having a bed of base-exchanging silicates or zeolites are given increased efficiency by producing occasional sudden pulsations in the liquid flowing through the bed, sufficiently energetic to cause a disturbance of the granular exchange materials.

Water-filter and silt trap. J. W. BRENKERT. U. S. 1,355,073, Oct. 5.

Disposing of garbage and other refuse. A. ENGLE. U. S. 1,354,733, Oct. 5. City refuse which may include night-soil, garbage, weeds and dead animals, is treated with  $\text{Ca}(\text{OH})_2$  and  $\text{H}_2\text{O}$  in a vault and allowed to undergo putrefactive decompn. Air is passed through the material and carries off  $\text{NH}_3$ , which may be recovered by passing it into an acid soln. or may be reacted upon by fumes from a S fire to cause formation of solid salts. The deodorized dried residue obtained may be used as a fertilizer

## 15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

J. J. SKINNER

Lime requirement of Pennsylvania soils. J. W. WHITE. Pennsylvania Agr. Expt. Sta., *Bull.* 164, 36 pp.(1920).—The lime requirement of 1474 samples of soil, from 50 counties and representing 98% of the soil area of the state, was detd. by the Veitch method. The well-drained soils are less acid than those poorly drained. No definite relation exists between the lime requirement and the texture of the soil. Most of the river bottom soils of the state are acid. The soils of the state which occupy the area where the av. lime requirement is from 0 to 1000 include the section where clover and alfalfa grow best. Most of the Volusia soils will not produce a good clover until the lime requirement is reduced below 5,000 lbs. A lime requirement of 3,000 lbs. will prevent a normal growth of clover in the Hagerstown soils. As a general av. clover fails on 49% of the unlimed soils of the state, produces a medium growth on 26% and a good growth on 15%.

J. J. SKINNER

The composition and origin of soils. F. F. H. CALHOUN. *Com. Fert.* 19, No. 5, 50, 52, 54(1919).—An address in which the following topics are considered: Elements used as plant food, elements found in common minerals, common minerals, common rocks, formation of soils, classification of soils, fertilizer requirements of soils and distribution of soils in South Carolina.

F. M. SCHERTZ

The influence of soil reaction on the growth of alfalfa. JACOB S. JOFFE. *Soil Sci.* 10, 301-7(1920).—Alfalfa was planted in pots containing soil to which varying amts. of  $\text{H}_2\text{SO}_4$  or  $\text{CaCO}_3$  were added. The hydrogen-ion concns. of the soils were detd. by Gillespie's method at the beginning, during and at the end of the growth. The soil varied from  $p_{\text{H}}$  3.0 to 7.1. The germination of alfalfa seed was practically the same in soils varying from  $p_{\text{H}}$  4.5 to 7.0 but was greatly reduced in soils of below  $p_{\text{H}}$  4.5. The yields of tops showed a gradual increase with an increase of  $p_{\text{H}}$  from 3.8 to 7.0. With increasing hydrogen-ion concn. the nodule production decreased. The N content of the plants showed a gradual increase with decrease in hydrogen-ion concn.

W. J. ROBBINS

Comparison of methods for determining soil acidity and a study of the effects of green manures on soil acidity. H. H. HILL. Virginia Agr. Expt. Sta., *Tech. Bull.* 19, 3-25(1919).—Expts. were conducted on 1 set of greenhouse pots and 2 sets of field plots. Deficiency of org. matter, prevalent in Virginia soils, may be overcome by green manuring; the acidity resulting therefrom is markedly influenced by alkalinity resulting from decompn. of plant ash. Legumes remove more Ca from the soil than non-legumes and in turning under the former the Ca returned to the soil exerts a certain effect in correcting acidity. The Jones method of estg. soil acidity is much more rapid than that of Veitch and applications of lime by its use have approached very near to actual field conditions; furthermore, it differentiates applications of  $\text{P}_2\text{O}_5$  on plots which produced corn over long periods. Pot expts. indicate that the incorporation of plant tissue produces a very slight increase in acidity; vigorous nitrate development was not inhibited. In field expts, with soy beans, buckwheat, red clover and rye, corn and wheat, no marked acidity was developed during a 5-yr. period. Green manuring in this case was not detrimental to a strong nitrate production, N fixation and plant growth. Field expts. where green crops were turned under every two weeks showed no injurious effects and after the second crop had been turned under the lime requirement diminished.

R. B. DEEMER

Nitrogen economy in the soil as influenced by various crops grown under control conditions. R. C. WRIGHT. *Soil Sci.* 10, 249-59(1920).—Leguminous and non-leguminous crops were grown in containers holding about 100 lbs. of soil and the N in the crop, N in the soil and N in fallow check soils and dry check soils detd. Under the conditions of the expt. the fallow soil showed a loss of N. Under certain crops there was an abs. loss of N in excess of that recovered in the crop and this varied with the crop and soil. Legumes may show this loss as well as non-legumes. When N fixation takes place in the growth of legumes the removal of the crop above ground depletes the soil of N just as if a non-leguminous crop had been grown. W. J. R.

Nitrate reduction in moist soil without the addition of energy material. ALICE ORLSNER. *Centr. Bakt. Parasitenk., Abt. II* 48, 210-21(1918).—In moist soils (about 40%  $\text{H}_2\text{O}$  content) denitrification occurs without the addition of C-containing sources of energy. The cause of this is a deficient supply of air. Every means of excluding air from soil bacteria increases the amt. of nitrate reduction. The nitrates are decompd. with the formation of elementary N. The source of energy for this denitrification process is the carbohydrate-containing organic material of the soil. Previously, denitrification in plant growth was considered from the viewpoint that the probability of the formation of free N from  $\text{KNO}_3$  by biological processes occurred only with the presence of much cellulose, straw and similar substances. But the above observations indicate that it is more likely to happen if the soil is poorly aired and if it is not especially rich in cellulose, straw, etc.

JULIAN H. LEWIS

Determination of cyanamide and dicyanodiamide in a sample of calcium cyanamide. MARQUERRE P. LORLETTE and L. DESVERGNES. *Ann. chim. anal. chim. appl.* 2, 164-7

(1920).—A review of Caro's, Kappen's and Ulipiani's methods of detg. these compds., with a critical discussion of errors present in them. A comparison is given of results obtained by L. and D. with these various methods. It is pointed out that volumetric methods based upon the detn. of the Ag in salts of cyanamide and dicyanodiamide give results which are inaccurate.

R. B. DEEMER

**The mechanism of the decomposition of cyanamide in the soil.** G. A. COWIE. *J. Agr. Sci.* 10, 163-76(1920).—C. finds upon treating soils with cyanamide under natural conditions and using amts. of cyanamide comparable with those used in practice, it is converted into urea by a purely chem. change and is then changed into  $\text{NH}_3$ . Ammonia does not result from the decompn. of cyanamide in sterile soils (heated to  $120^\circ$  or  $135^\circ$ ), but the addition of soy bean results in the production of considerable amts. of  $\text{NH}_3$ . It was proven that urea was formed when cyanamide was added to sterile soils, which persists as such because of the suppression of the necessary urea-decomposing organisms. With soils heated to  $100^\circ$  urea is first produced, which then decomposes into  $\text{NH}_3$  after the recovery of the appropriate organisms. In unheated clay and sandy soils appreciable amts. of urea were formed during the initial periods of treatment, but the production of  $\text{NH}_3$  was rapid and progressive. Expts. with cyanamide and ordinary impure quartz sand show that urea is not produced, and cyanamide does not appear to break down in the manner above indicated in peat soils; in these it gives rise to a relatively small amt. of urea under normal conditions. "The investigation has not revealed the exact nature of the decomp. agent in the soil. It is interesting to note, however, that a sample of Thanet sand taken from a boring through the London Clay near Chelmsford was found even after ignition to be active in decomp. cyanamide into urea. This particular sand has been shown to contain a constituent resembling a zeolite in being reactive and possessing property of softening hard  $\text{H}_2\text{O}$  by the substitution of Na salts and possibly K for those of Ca and Mg. In following up this clue it was found that the addition of a definite zeolite, prehnite, to ordinary inert sand produces a mixt. capable of converting cyanamide into urea."

R. B. DEEMER

**Sulfur in relation to soil.** W. L. POWERS. *United States Reclam. Rec.* 11, 28-9 (1920).—Improved methods of analysis show that alfalfa, per crop of 5 tons, removes about 35 lbs. of S per acre and about 25 lbs. of P. Rape contains 20 lbs. of S per ton. The paper briefly reviews the work done on S fertilizing and S removed by average crops in the states of Wisconsin, Kentucky, Iowa and Oregon. In Oregon, increases as great as 500% have been obtained and never have any injurious effects been noted, from heavy applications of S.

F. M. SCHERTZ

**Sulfur as a fertilizer.** CHAS. A. SHULL. Univ. Kentucky. *Science* 52, 376-8 (1920).—It is probable that S is just as often a limiting factor of crop growth as is P, N or K. When S is a limiting factor the addition of any other fertilizer is useless. So, too, when Ca or Mg are limiting factors. Fertilizer expts. which have been made comparing acid phosphate with bone meal,  $\text{K}_2\text{SO}_4$  with KCl, or  $(\text{NH}_4)_2\text{SO}_4$  with  $\text{NaNO}_3$  have at least two variables if S itself is an important fertilizer element and are therefore invalid for the purpose of their comparison as sources of P, K or N. "It is highly important that our fertilizer practice should be put on a rational basis."

ALBERT R. MERZ

**Coöperative experiments for the composing of phosphate rock and sulfur.** W. B. ELLERT AND W. G. HARRIS. *Soil Sci.* 10, 315-25(1920).—Detns. were made of the water-sol., ammonium-citrate-sol., and total  $\text{H}_4\text{PO}_4$ , as well as the  $\text{H}_2\text{SO}_4$ , total N,  $\text{NO}_3$  and  $\text{NH}_3$  in composts of soil and rock phosphate; soil, rock phosphate and sulfur; soil, rock phosphate, sulfur and manure, uninoculated and inoculated with sulfifying microorganisms. The results show that the addition of manure to a compost

of soil, S and rock phosphate increases the availability of the rock phosphate to a greater extent than S alone. Without S no appreciable increase in the availability of the rock phosphate was noted. All Virginia soils have some sulfofying power. The addition of phosphate to manure decreased the rate of fermentation and resulted in 2 years in a loss of 57.8% of the dry matter and 48.2% of the N. S and phosphate together decreased the rate of fermentation still further. The composting of S, soil, rock phosphate and manure is not recommended for the use of farmers because of the slowness of the process and mechanical difficulties.

W. J. ROBBINS

The influence of initial reactions on the oxidation of sulfur and the formation of available phosphates. J. G. LIPMAN AND J. S. JOFFE. *Soil Sci.* 10, 327-32(1920).—To soil containing 15% rock phosphate and 5% S varying quantities of  $H_2SO_4$  were added. The hydrogen-ion exponent of the soils varied from  $pH$  4.7 to 5.4. The sol. phosphates and hydrogen-ion concns. were detd. weekly. Within one week the  $pH$  had fallen to 3.6-4.4, in 3 weeks to 2.4-2.8, and by the end of the 12th week to 1.4-2.0. Little further change occurred. The amt. of sol. phosphate increased during the 20 weeks of the expt., reaching 83 to 85%, but no influence of the initial reaction on the formation of the sol. phosphate was noted.

W. J. ROBBINS

The manurial value of bracken. F. T. SHUTT. *Agr. Gazette Can.* 6, 328-9(1919).—The distribution of the common bracken or brake (*Pteris aquilina* L.) in Canada is given and from the analysis of 2 samples S. points out the value of the fertilizing materials present. The percentages of constituents of fertilizer value in these were as follows: N, 1.29, 1.84;  $P_2O_5$ , 0.43, 0.68;  $K_2O$ , 1.52, 2.75. General statements are made but no exptl. evidence is offered.

R. B. DEEMER

Manurial experiments with sea island cotton in St. Vincent in 1918-1919, with some notes on the control of certain diseases by spraying. S. C. HARLAND. *West Indian Bull.* 18, 20-31(1920).—Expts. of 7 years duration are discussed in detail; a summary of the main features is as follows: Cotton grown continuously for a number of years on the same land responds to the application of both artificial and org. fertilizers. The characteristic sign of potash hunger now appearing indicates the need of potash fertilizers. The use of phosphate as a fertilizer is not recommended, as potash and phosphate are inferior to potash alone. Cotton-seed meal alone is not economical. Time of maturity is not affected by use of manures.

R. B. DEEMER

Possibilities of Cambodia from the cotton grower's standpoint. M. DE FLACOURT. *General Govt. of Indo-China. Congrès d'Agriculture coloniale organisé par l'Union coloniale française. Série Saigon. Bull.* 5, 29 pp.(1918).—A red soil (Chambak-Meas field) and a gray soil (Thbong-Krapoen field), resp., analyzed per 1,000 of soil dried at 100°: Siliceous gravel, ———, 38.500; sand, 313.60, 842.913; limestone, 2.02, 0.707; clay, 645.98, 101.919; org. debris, 38.40, 15.961; humus, trace, traces; N 1.428, 0.258;  $P_2O_5$  3.843, 0.596;  $K_2O$  0.373, 0.733; CaO 1.133, 0.396; MgO 0.150, 0.192.

ALBERT R. MERZ

Analysis of droppings of caterpillar (*Antheraea cytherea*). CHARLES F. JURITZ. *Chem. News* 121, 181(1920).—This caterpillar in certain years completely strips the wattle (*Acacia cyclops*) of its leaves and the droppings become so abundant that their use as fertilizer has been proposed. Analysis gives  $H_2O$ , 11.10%; ash, 11.06%; N, 2.07%;  $K_2O$ , 2.87%; CaO, 1.86% and  $P_2O_5$ , 0.87%. Leaves of the *A. cyclops* analyze  $H_2O$ , 71.96%; ash, 1.39%; N, 1.07%;  $K_2O$ , 0.25%; CaO, 0.13% and  $P_2O_5$ , 0.12%. Comparison is made of the compn. of the droppings with that of horse and cow manure and of kraal manure. (*Report S. African Assoc. Adv. Sci.* 1915, p. 142.)

ALBERT R. MERZ

Some observations upon the effect of Borax in fertilizers. W. J. MORSE. *Maine Agr. Expt. Sta., Bull.* 288, 89-120(1920).—A summation of conditions as found in the

potato fields of Maine in 1919, where fertilizers containing borax were used. It was ascertained that the potato crop failure appeared to be associated with the use of fertilizers containing K. No injury was produced where fertilizers containing no K were used. B was found present in appreciable amts. in the K-containing fertilizers which were used on those fields where the type of injury in question appeared. No definite cases of similar injury were observed where it could be shown that borax-free fertilizers carrying similar amts. of N, P and K were applied. On fields where borax was used, there was a stunted appearance of the plants with a large number of misses. The foliage of the injured plants had a characteristic appearance. There was considerable yellowing of the leaves, more particularly of the margins. This was prominent on the more dwarfed and more severely injured plants. The yellowing was of a bright golden color, and not the pale sickly yellowing usually present in plants that are normally or prematurely ripening. In mild cases the abnormal color was restricted to the extreme edges of the leaves. In severe cases the leaves were frequently narrowed and often folded upwards on the mid-rib. Pot expts. were made in which fertilizers containing borax were applied to potatoes, beans, oats, wheat and buckwheat. Fertilizers containing borax were applied in varying amts. so that the amt. of  $\text{Na}_2\text{B}_4\text{O}_7$  used varied from 0 to 38.6 lbs. per acre. The results of the greenhouse expts. to a large extent confirmed the field observations. All plants receiving B were affected. Killing of the tips and margins of the leaves was characteristic. The large applications caused severe root injury. An application of fertilizer in the drill equivalent to 4.4 lbs.  $\text{Na}_2\text{B}_4\text{O}_7$  per acre caused severe injury to beans, while broadcast-ing the same fertilizers, applying the equiv. of 8.8 lbs., caused no apparent injury to oats, wheat and buckwheat.

J. J. SKINNER

Injury to crops by borax. J. K. PLUMMER AND F. A. WOLF. North Carolina Dept. Agr., *Bull.* 275 (Vol. 41, No. 15), 20 pp.(1920).—An account is given of the injury in N. Carolina by fertilizers containing borax in 1919, together with a description of the character of injury to tobacco, cotton, corn and peaches. Investigations in pots with corn and cotton on Cecil clay loam and cotton and tobacco on Durham sandy loam agrees with field observations in showing that plants are more susceptible to injury on the lighter soils. In sandy soils, as little as 1 lb.  $\text{Na}_2\text{B}_4\text{O}_7$  per acre injured tobacco and no cotton grew where over 5 lbs. was used. In clay soil cotton and corn showed marked injury when the amts. of  $\text{Na}_2\text{B}_4\text{O}_7$  exceeded 7 lbs. per acre. Corn plants on sandy soil, to which borax at the rate of 5 lbs. per acre was applied showed marked injury, and with 10 lbs. they were lacking in green color and soon died. Interference with color formation by B does not appear to be related to the assimilation of Fe nor to plastid formation. In nutrient soln. growing wheat, as little as 5 p. p. m. cause discolored foliage and 100 p. p. m. interfered with proper root development. In agar solns. 5 p. p. m. slightly checked plant development.

J. J. SKINNER

The province of industrial explosives in agriculture. ALFRED STETTbacher. *Z. angew. Chem.* 33, I, 203-4(1920).—An appeal for the practical use of explosives in a scientific manner.

C. G. STORM

Miscellaneous soil insecticide tests. JOHN J. DAVIS. *Soil Sci.* 10, 61-75(1920).—See C. A. 14, 3495.

W. J. ROBBINS

Experiments on the toxic action of certain gases on insects, seeds and fungi. I. E. NEUFERT AND G. L. GARRISON. U. S. Dept. Agr., *Bull.* 893, 1-16(1920).—The gases used in this investigation were illuminating gas, CO, HCN,  $\text{AsH}_3$ , phosgene, cyanogen chloride and chloropicrin. Only the last 2 gases mentioned give promise of being useful for fumigation purposes. Neither of these, however, can be used in greenhouse fumigation because of their injurious action on plants, but they can prob-

ably be used in the fumigation of stored products. Chloropicrin has undoubted efficiency as an insecticide, and in general is more poisonous to stored-product insects than HCN. The effect of cyanogen chloride as an insecticide is practically the same as that of HCN.

W. H. ROSS

**Burgundy mixtures and other copper sprays.** G. T. SPINKS. Univ. Bristol. *Ann. Rept. Agr. Hort. Res. Sta.* 1918, 19–24.—A study of various amts. of soda to be used with  $\text{CuSO}_4$  in these sprays resulted in bringing out the following points: Mixts. having a ratio of soda to  $\text{CuSO}_4$  of 4:4.25 kept for the longest time in a condition fit for spraying, while mixts. having the ratio of 4:5 crystallized most rapidly. Mixts. having a ratio of 4:4.25 and 4:5 have equally good covering qualities, while the 4:7.2 mixt. is slightly inferior. All of these mixts. adhere and resist rain equally well, and all are equal as regards the possibility of scorching foliage. A unique method of testing these sprays in the laboratory for their effectiveness as fungicides is given in detail, and from these tests the indications are that all the mixts. are equally effective, but as yet no satisfactory field tests have been made.  $\text{CuSO}_4$  and soda crystals finely powdered and mixed together and then dissolved give solns. equal in every respect to those prepared in the usual way. Further tests on a Cu stearate spray indicate that its fungicidal properties are as good as the Burgundy mixts.

R. B. DEEMER

**The influence of chemical constitution on the toxicity of organic compounds to wireworms.** F. TATTERSFIELD AND A. W. R. ROBERTS. *J. Agr. Sci.* 10, 199–232 (1920).—Twenty-five org. compds. were studied and their order of effectiveness is given. In general the effect of a group of compds. of the same type is directly detd. by the chem. constitution of the type. The particular effects of individual members are limited by the physical properties such as volatility, etc. It is worthy of particular note that a limit is put upon toxicity by decrease in vapor pressure, when it sinks too low to allow a toxic concn. in the vapor phase. Chem. inert compds. boiling above  $170^\circ$  are generally uncertain in the effect on wireworms after an exposure of 1000 min. at  $15^\circ$ . Nearly all org. compds. boiling above  $215^\circ$  are uncertain in their action, while those boiling above  $245^\circ$  are non-toxic. The limits depend on the resistance of the insect, the length of the exposure and the temp.

R. B. DEEMER

**Making Bordeaux mixtures and some other spray problems.** W. S. FIELDS AND JOHN A. ELLIOTT. Arkansas Agr. Expt. Sta., *Bull.* 172, 12 pp. (1920).—Eight methods of mixing Bordeaux were studied. For a stock soln., 1 lb. of  $\text{CuSO}_4$  was used to 1 gal. of  $\text{H}_2\text{O}$  and 1 lb. of freshly slaked stone lime with the same amt. of  $\text{H}_2\text{O}$ . The best results were obtained by making a weak Cu and strong lime soln. which consisted of 1.6 gal. of stock  $\text{CuSO}_4$  soln. dild. with 16.8 gal.  $\text{H}_2\text{O}$  poured into 1.6 gal. of stock lime soln. The resulting soln. showed less pptn. and less spherocrystals than the other mixts. used. Water hardness was found to have no appreciable effect upon the pptn. of Bordeaux or Bordeaux-lead-arsenate mixts., or sedimentation of lime-sulfur-lead-arsenate mixts. The amt. of As in soln. when either acid or neutral lead arsenate is used is small.

J. J. SKINNER

Effect of salts of boron upon the distribution of desert vegetation (KELLERMAN)  
11D.

OLARU, D. A.: Role du manganèse en agriculture. Paris: Baillières et fils.  
For review see *Bull. soc. hyg.* 8, 500(1920).

**Fertilizer.** A. C. BOHRER. U. S. 1,354,719, Oct. 5. A fertilizer is formed of an insol. phosphate such as "Cafsa-phosphate," "Florida hard rock" and apatite mixed with urea nitrate which will form sol. phosphate in contact with moisture in the soil.

**Fertilizer.** F. S. WASHBURN. U. S. 1,355,369, Oct. 12. Crude  $\text{CaCN}_2$  is treated with  $\text{H}_2\text{O}$  and the soln. thus formed is reacted upon with  $\text{H}_3\text{PO}_4$  to form a fertilizer.

**Fertilizers.** CHEMISCHE FABRIK RHEINANIA AKT.-GES. and G. A. VOERKELIUS. Brit. 148,242, July 9, 1920. To obtain a fertilizer containing phosphates, N and potash, phosphates are treated with  $\text{HNO}_3$  and an equiv. amt. of  $\text{K}_2\text{SO}_4$  is added to prevent the formation of deliquescent  $\text{Ca}(\text{NO}_3)_2$  and to obtain a higher % of sol. phosphates. Phosphates containing Fe and clay can be used for making superphosphates by this method, which also allows of the use of more dil.  $\text{HNO}_3$ .

**Fertilizers.** CHEMISCHE FABRIK RHEINANIA AKT.-GES. and G. A. VOERKELIUS. Brit. 148,243, July 9, 1920. Addition to 148,242 (above). In obtaining a non-deliquescent fertilizer by the action of  $\text{HNO}_3$  on phosphate as described in the principal patent, the  $\text{K}_2\text{SO}_4$  added therein may be replaced or supplemented by  $\text{K}_2\text{SO}_4\cdot\text{MgSO}_4$  or by  $(\text{NH}_4)_2\text{SO}_4$  mixed with  $\text{K}_2\text{SO}_4\cdot\text{MgSO}_4$ ,  $\text{K}_2\text{SO}_4$ , or both. The quantity of sulfate added may be reduced to an amt. equiv. to from  $1/3$  to  $2/3$  of the  $\text{HNO}_3$  used, which itself need only be added in sufficient quantity to insure an excess of acid of from 0-1%. The strewing properties of the product obtained are improved by mixing the sulfate first with the acid or the phosphate, and then adding the mixt. to the third component.

**Fertilizers.** H. DEVOS. Brit. 147,798, July 9, 1920. A fertilizer is obtained by heating waste leather and other animal detritus in a boiler or autoclave with dil. acid or acid salt, preferably  $\text{H}_2\text{SO}_4$ , at about  $95^\circ$ , boiling, agitation, or evapn. of the liquid being avoided. After 1-4 hrs., a muddy mass collects at the bottom and the supernatant acid liquor is decanted off, and may be used again. The muddy mass is allowed to solidify on slabs, dried in the air on screens, and after some days pulverized in a crusher.

**Fertilizer from peat.** W. B. BOTTOMLEY. U. S. 1,355,732, Oct. 12. Auximones are produced in peat by first developing a nucleic acid deriv. in the peat by moistening it with a 2-10% soln. of  $\text{NaCl}$  or similarly acting non-alk. salt, heating the moistened peat to about  $100^\circ$  while retaining the moisture in it and then maintaining the peat under conditions favorable to the growth of microorganisms.

**Fertilizer from carnotite tailings.** E. MILLER. U. S. 1,353,931, Sept. 28. A fertilizer suitable for general use is prepd. by heating (to  $350\text{--}400^\circ$ ) and grinding tailings of crude carnotite ore and mixing with other material such as peat, sawdust, coal dust or lime.

**Inoculation of soil and seeds.** C. F. DILLER. U. S. 1,354,808, Oct. 5. A prepn. of soil organisms for treating soil or seeds is prepd. by absorbing a tested culture in a previously dried and pulverized inorg. solid such as silica, K silicate,  $\text{CaCO}_3$  or phosphate rock in the proportions of 25 cc. of the liquid to about 100 g. of the solid material. Such a prepn. is stated to be capable of remaining active for several years.

**Weed-destroying mixture.** G. KRAMER. U. S. 1,354,043, Sept. 28. A mixt. for killing weeds is formed of lye 10,  $\text{NaCl}$  10,  $\text{KNO}_3$  20 and slack coal 40 parts.

## 16—THE FERMENTATION INDUSTRIES

H. S. PAINE

**Industrial alcohol.** BURNELL, R. TUNISON. *J. Franklin Inst.* 190, 373-420 (1920).—Full information is given concerning the history, raw materials, mode of manuf., consumption, uses and future of industrial alc. Cf. *C. A.* 14, 1404.

JOSEPH S. HEPBURN

**Production of alcohol from Jerusalem<sup>\*</sup> artichokes.** M. RÜDIGER. *Z. Spiritusind.* 43, 203(1920); *J. Soc. Chem. Ind.* 39, 608A.—Lab. expts. indicate that a fair yield of

alc. could be obtained from Jerusalem artichokes in small distilleries. Tubers which had remained in the ground until the new year, and had thus deteriorated, gave a yield of 6.4 l. of alc. per 100 kg. by fermentation of a mash of the raw pulped material without preliminary hydrolysis. By heating the mash for an hour at 55° before fermentation the yield of alc. was increased to 0.4 l., but heating at 75° or 100° reduced the yield. Higher yields were obtained by heating with acid, but this would not be economical for small distilleries. The spirit obtained, carefully distd. once, was more palatable than spirit from beets.

W. B. V.

Preparation of alcohol from wood. EMIL HEUSER. *Cellulosechemie* 1, No. 5, 41-6(1920).—A lecture.

C. J. WEST

Wood as raw material for alcohol production. F. KORITSCHNER. *Pharm. Monatshefte* 1, 93-7(1920).—A review of various raw materials available for the production of alcohol, including some expts. on the fermentation of products resulting from the hydrolytic treatment of wood shavings with  $H_2SO_4$ .

W. O. E.

Motor alcohol from molasses. J. F. CHILD. *Louisiana Planter* 64, 381-2(1920).—A discussion of the use of alc. as a motor fuel and also of the use of alc.-ether mixts. A description is given of the app. and methods used in production, with yields.

C. H. CHRISTMAN

Preparations for working corn (in distilleries). FOTH. *Z. Spiritusind.* 43, 307 (1920).—A review of changes in plant and in operation necessary for the conversion of distilleries from a potato to a corn raw material, in expectation of the removal of restrictions on the use of the latter.

W. B. V.

Fermentation in open and closed fermenting tubs. E. LÜNDER. *Z. Spiritusind.* 43, 276(1920).—It has often been observed that mash fermenting in closed iron vats show a rapid temp. rise at the beginning of the principal fermentation, and a rapid drop in temp. at the end of that period. The explanation of the first phenomenon is thought to be the greater vol. commonly used in iron vats than in the older open wooden ones, and the lack of air cooling at the surface; the subsequent drop in temp. is probably due to the greater cond. of the thin sheet-iron walls. The present investigation was designed to det. especially the influence of these temp. differences on the rate of yeast multiplication. Rye and barley worts were fermented by 1 gram per liter of a brewer's yeast ("Rasse M") in glass flasks, containing 1 to 3 liters, some of which were open and some closed by a 10 cm. water seal, all being held in a water bath whose temp. could be changed easily. From time to time the flasks were shaken vigorously and samples were withdrawn by means of a pipette. These were diluted, and 30 cell counts made from each of six samples, so that each result is the av. of 180 counts. The seed yeast was counted after an equiv. dila. in distd. water. The temp. of pitching was 17°, that of fermentation, after about 30 hrs., 28°; the latter temp. was maintained until the end of the expt. Three series of expts. resulted similarly with approx. the following yeast growth: in open flask, 24 hrs., 8-fold; 48 hrs., 17-fold; in closed flask, 24 hrs., 15-fold; 48 hrs., 17-fold; 72 hrs., 17-fold. In each case the closed vessels showed slightly higher alc. content at the end than the open vessels. The increased yeast growth during the first 24 hrs. in the closed vessels is ascribed to more rapid temp. increase, rather than to contact with the air, partly because of the known advantages of aeration in the com. manuf. of yeast.

W. B. V.

Relation between the organic acids of wines and tartaric acid (Issoglio). VERDA. *Schweiz. Apath. Ztg.* 58, 193-5(1920); cf. C. A. 13, 2724.—A résumé of a paper by Issoglio, *Ann. dell'Accad. dell'Agric.* To ascertain addition of tartaric acid (T) to wines, so as to mask dila. with  $H_2O$ , I. proposes for Italian wines the following detns.: (1) Total acidity, A = no. of cc. of N NaOH to neutralize the free acid in 1 l. of wine. (2) Alkalinity of ash, B = no. of cc. of N  $H_2SO_4$  to neutralize total ash alkalinity



from 1 l. of wine; (3)  $T$  (Halenke-Möslinger method),  $C$  = cc. of  $N$  soln. required to neutralize the  $T$  of 1 l. of wine. The relation will then be:  $r = [(A + B) - C]/C$ . In detg.  $r$  for 76 Piedmontese wines, I. found an av. of  $r = 2.58$  (1.50 to 3.50 in 70 wines). The formula must be tested by further analyses of authentic Italian wines; the keeping qualities of the org. acids during definite periods must also be studied. When  $T$  has been added to a wine, ( $x$ ) another  $r$ , *i. e.*,  $r'$  is found, and from the observed deviation  $d = r - r'$ , the amt. of  $x$  is calcd. by the formula:  $x = dC^2/(A + B - C - dC)$ . When  $T$  ( $C$ ) diminishes, as *e. g.*, in the plastering or in chaptalizing of wines,  $r$  increases notably; for a moderately plastered wine,  $r$  was found = 11.8; in a wine that had soured,  $r$  was = 37.7.

S. WALDBOTT

Dealcophilized malt beverages. A. L. STRAUS. U. S. 4,354,585, Oct. 5. A mixt. for restoring the taste to malt beverages from which the alc. has been boiled off is formed of NaCl 10 lbs., a foam-producing substance such as gum arabic 10 lbs., a sweetening material such as sugar 3 lbs., K metabisulfite 1 lb., citric acid 1 lb. and lupulin, tannin or quassia 2 oz.

## 17—PHARMACEUTICAL CHEMISTRY

W. O. EMERY

War products. WALTER DULIÈRE. *J. pharm. Belg.* 2, 873-6(1920).—Attention is directed to the widespread sale of spurious chemicals and drugs in Belgium since the war. Neosalvarsan, fluidext. of cinchona, ext. of opium, cod liver oil and pyramidone are the items receiving special mention. Some samples of the fluidext. of cinchona and of the ext. of opium have been found to contain only traces of alkaloids, seal oil is often sold for cod liver oil and pyramidone is adulterated with a mixt. of potato or rice starch and Mg silicate. Nine samples of pyramidone in powdered and tablet form were analyzed.

A. G. DuMEZ

*Polygonum bistorta* L.; localization of tannin; its use as a substitute for *Krameria triandra*. RUIZ AND PAVON. *J. pharm. Belg.* 2, 876-8(1920).—A proximate analysis of the rhizome showed the following: water, 6.3%; ash, 4.35%; tannic acid, 18.25%; reducing sugars, 2.13%; gums and mucilages, 2.23%; starch, resin and cellulose, 66.74%. By the direct contact or osmotic method, using ammonium molybdate as the reagent, the presence of tannin was established in all tissues, but was found to predominate in the cortical parenchyma. It is concluded that the rhizome is a satisfactory and economical substitute for *krameria*.

A. G. DuMEZ

Adulteration of neosalvarsan. M. F. DEMYTTENAERE. *J. pharm. Belg.* 2, 853-6(1920).—Eight samples of neosalvarsan substitutes of German origin were examined. One consisted of  $\text{NaHCO}_3$  colored yellow with an unidentified organic substance, 3 were composed of NaCl and  $\text{BaCrO}_4$ , 2 were wheat flour colored with  $\text{PbCrO}_4$ , 1 was a mixt. of  $\text{Na}_2\text{SO}_4$  and  $\text{CaSO}_4$  colored with  $\text{PbCrO}_4$ , and 1 was a slightly deteriorated specimen of the genuine product. Tests recommended by the manufacturers of novarsenobenzene for establishing the identity and purity of the product are given in detail, also the method of C. N. Myers and A. G. DuMez for the detn. of the As content.

A. G. DuMEZ

Matriculation sheets. A. SCHAMMELHOUT. *J. pharm. Belg.* 2, 630, 652, 698, 719, 761, 777, 791, 792, 824(1920).—Tentative monographs proposed for introduction into the Belgian National Formulary are submitted for the following items: methyl-atropine bromide, brucine, Bi, bismuth subcarbonate,  $\text{CdSO}_4$ ,  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{BiO}_3\text{H}$ ,  $\text{BiOI}$ , and  $\text{Bi}(\text{NO}_3)_3 + 3\text{H}_2\text{O}$ .

A. G. DuMEZ

Supplement to the French pharmacopeia. A. SCHAMMELHOUT. *J. pharm. Belg.* 2, 645-7, 668-8, 693-5, 709-11, 733-5, 749-51, 769-70, 785-6(1920).—A review of the new supplement to the French pharmacopeia which became official July 1, 1920.

A. G. DuMEZ

Hypophysial extract. E. HERCOT. *Chem. & Drug.* 93, 65-6(1920).—A critical review of methods which have been proposed for the physiol. standardization of hypophysial ext.

A. G. DuMEZ

Examination of arsphenamine and neoarsphenamine. URZ. *Süddeut. Apoth.-Ztg.* 60, 187-8(1920); *Chem. Zentr.* 1920, II, 772.—Arsphenamine (A) preps. may be identified by the fact that they give the diazo reaction with  $\alpha$ -naphthylamine but not with  $\beta$ -naphthylamine. To distinguish between A and neoarsphenamine (B) the following reactions may be employed: 5 cc. of B soln. (1:1000) gives a ppt. with 0.1 cc. of dil HCl which dissolves on boiling and does not reppt. on cooling. A does not give a ppt. under these conditions. B solns. give a cloudiness changing to an orange-colored ppt. on heating with an equal vol. of AcOH. A gives a negative result. Both preps. give a green to red coloration with FeCl<sub>3</sub> soln. With an excess of this reagent A soln. becomes dark red and B violet. HgCl<sub>2</sub> gives a heavy white ppt. with A and an orange ppt. with B. Both become gray and dirty in color and rapidly sink to the bottom on heating. With NH<sub>4</sub> molybdate A gives an olive-green ppt. and B a dark green coloration, but no ppt. H<sub>2</sub>PtCl<sub>6</sub> gives a flocculent ppt. with B which gradually settles. No ppt. is formed with A. Picric acid gives an intense yellow ppt. with A solns. which is not dissolved on heating. Only a slight opalescence is produced in B solns. after long standing. A solns. are changed to a grayish green followed by a gray cloudiness on the addition of concd. NaHSO<sub>4</sub> soln. On warming, the liquid becomes green and then yellow and finally a lemon-yellow ppt. forms. B gives no reaction.

A. G. DuMEZ

Determination of hydrastine in *Hydrastis canadensis* and its preparations by means of silicotungstic acid. N. WATTIEZ. *J. pharm. Belg.* 2, 817-9(1920).—Place 7.5 g. of the powdered rhizomes, previously dried at 50°, in a 250-cc. round-bottom flask equipped with a reflux condenser, add 150 g. of EtOH (70%) and digest on a water bath for 3 hrs. Replace the EtOH lost by evapn., shake vigorously, allow the mixt. to cool and filter. Collect 120 g. of the tincture (6 g. of powd.), evap. to a sirup and ppt. the berberine by adding sufficient H<sub>2</sub>SO<sub>4</sub> (25%) to bring the total wt. to 15 g. After 1 hr., add 1 g. of talc and filter through a folded filter (8 cm.). Collect 10 g. of the filtrate in a 125-cc. tared erlenmeyer, make alk. with 5 or 6 cc. NH<sub>4</sub>OH, cool, add 40 g. of Et<sub>2</sub>O and agitate frequently during 1/2 hr. Allow the mixt. to sep. and decant 35 g. of the ethereal layer (3.5 g. powder). Wash the ethereal soln. with distd. H<sub>2</sub>O and ext. with HCl (1%) until the aq. ext. remains clear when tested with Mayer's reagent. Unite the aq. exts., filter, heat to remove the dissolved ether, cool and ppt. the hydrastine by adding 15 cc. of silicotungstic acid soln. (5%) and heating to boiling. Allow the mixt. to stand for 24 hrs., collect the ppt. on a filter, wash with H<sub>2</sub>O until free from acid and dry to const. wt. The wt. multiplied by 0.534 gives the amt. of hydrastine in 3.5 g. of the powder. In the assay of the fluidextract of hydrastis, 6 g. is taken and in the case of the tincture 30 g.

A. G. DuMEZ

Rapp's method for the determination of alkaloids. A. HEIDUSCHKA AND L. WOLF. *Süddeutsch. Apoth.-Ztg.* 60, 142-3(1920); *J. Soc. Chem. Ind.* 39, 465A.—See C. A. 13, 3273.—In the case of *nux vomica*, evap. 100 g. of the EtOH-CHCl<sub>3</sub> soln. of the alkaloids to a small bulk, add 25 cc. of CHCl<sub>3</sub> and shake with 20 cc. of 2% H<sub>2</sub>SO<sub>4</sub>. After clarification, mix 14 cc. of the clear filtrate with 3 cc. of 15% NaOH and shake for 3 mins. with 70 cc. of CHCl<sub>3</sub> and 25-30 g. of plaster of Paris. Filter 60 cc. of this soln. after clarification and shake with 12 cc. 0.1 H<sub>2</sub>SO<sub>4</sub>. Finally dil. with 12 cc. of

H<sub>2</sub>O and titrate 20 cc. of this mixt. with 0.1 N NaOH. The percentage of alkaloid is equal to (10 cc. 0.1 N NaOH) 3.64 + 5.

A. G. DUMÉZ

Preparation of tinctures. BODINUS. *Apoth.-Ztg.* 24, 436(1919).—A reply to to Anselmino's criticism of B.'s procedure (cf. C. A. 13, 2966).

W. O. E.

Eucalyptol determination. C. T. BENNETT AND M. S. SALAMON. *Perf. Essent. Oil Rec.* 11, 302(1920); cf. C. A. 14, 1001.—A recently suggested method by Cocking was found to possess no advantage over that now official in the Br. Pharm. The authors find after repeated tests that the constituents in eucalyptus oil, other than the eucalyptol, have a marked influence on the m. p. of the cresol compd., and that therefore this test is only useful as a rapid works method for grading oils from the same variety of eucalyptus.

W. O. E.

*Rhus venenata* DC. HEBER W. YOUNGKEN AND GEORGE A. SLOTHOWER. *Am. J. Pharm.* 92, 695-701(1920).—A study of the histological nature of the stems and leaves of *Rhus venenata* DC., and observations on its poisonous constituent. The dried leaves and petiole yielded a dark green-colored powder with a very peculiar, offensive odor. On ignition 6.5% ash was obtained. The residue was grayish brown and had an alk. reaction. Traces of K, Cl, PO<sub>4</sub> and SO<sub>4</sub> were found in the ash. Tests for nitrates and nitrites were negative. The poisonous principle in the form of non-volatile fixed oil was obtained by extn. with a good grade of low-boiling petroleum ether. On standing a white waxy like substance sepd. out of the petroleum ext. By spontaneous evapn. of a portion of the petroleum ext. a yellowish brown, oily residue was obtained. This residue, when taken up with neutral alc. is neutral in reaction. The alc. soln. of the residue was optically inactive, which Pfaff states is true of the non-volatile oil causing the dermatitis.

W. G. GARSSLER

Gum vereck, arabic or Senegal in Anglo-Egyptian Soudan. E. PERROT. *Bull. sci. pharmacol.* 27, 465-74(1920).—A description of the methods of production of gum arabic.

F. S. HAMMETT

Secalopan: a new preparation from *Secale cornutum*. FR. UHLMANN AND U. MIRMELSTEIN. *Z. exp. Path. Ther.* 21, 37-57(1920).—The new prepn. is by far the most active substance of all the commercially prepd. compds. of similar action studied. Ergotone gives a slightly greater stimulus to the isolated intestine which is attributed to the greater concn. of the latter drug. Although secalopan is more active its toxicity is not greater than the majority of the com. secal prepn. It acts to a lesser degree on the blood vessels and blood pressure and is less irritating on subcutaneous or intramuscular injection than the other prepn.

F. S. HAMMETT

Some of the important constituents of digitalis. WILLIAM J. MCGILL. *J. Am. Chem. Soc.* 42, 1893-1900(1920).—This work was carried out in order to det. if it is possible to obtain fractions of uniform compn. by using the same method of procedure on different samples of the drug, and if so just which constituents will be present in a given fraction. Pittenger's goldfish method (C. A. 14, 594) was used in testing the relative values of the different fractions. It was found that with careful manipulation and uniform methods of procedure it is possible to obtain crude fractions, by means of various solvents, of a reasonably const. chem. compn. The cold H<sub>2</sub>O ext. of the leaves contains either no or a very minute amt. of digitoxin, its activity being due entirely to a CHCl<sub>3</sub>-sol. and a CHCl<sub>3</sub>-insol. fraction. The saponin substance of the leaves, digitasaponin, is a totally inactive and non-hemolytic substance when purified. Hatcher's CHCl<sub>3</sub>-sol. fraction (C. A. 14, 595) of the infusion is a mixt. of "gitalin" and digitoxin.

CHAS. A. ROULLER

Theobromine determination in sodium salicylate-sodium theobromine (diuretin). O. P. A. H. SCHAAP. *Wormerveer. Pharm. Weekblad* 57, 1234-6(1920).—In the Vulpius method (Holland Pharm. IV), pptn. of theobromine from diuretin is best done

with  $\text{NH}_4\text{Cl}$  instead of the prescribed separate addition of dil.  $\text{HCl}$  and  $\text{NH}_4\text{OH}$ . In either case, however, the method is very inaccurate (error about 14%). Accurate detns. may be made as follows: Treat 1 g. of diuretin in 5 cc. of  $\text{H}_2\text{O}$  with 5 cc. of neutral  $\text{ZnSO}_4$  soln. (1:10), boil a few min., cool, filter, wash free from sulfate and salicylate with cold  $\text{H}_2\text{O}$  and dry at  $100^\circ$  to const. wt. Further studies will be made with a view to applying this method to the quant. examn. of cocoa, chocolate, etc.

JULIAN F. SMITH

**Cinchona assay.** F. LEHMANN. *Arch. Pharm.* 258, 85; *Pharm. J.* 105, 287 (1920).—Of the 2 methods in use, that with dil. aq. acid probably includes all the cryst. cinchona alkaloids; that with alkalies and org. solvents gives a less pure alkaloid, but a larger yield. Apparently, the bark contains 2 kinds of alkaloids differing in soly. An ext. prepd. by the 2nd process gives too low results when assayed on the basis of the 1st. The Swiss Pharm. in prepg. the fluid ext., directs exhaustion with  $\text{EtOH}$ , which exts. both acid-sol. and insol. alkaloids. To assay the ext., mix 2.5 g. with 2 g. dil.  $\text{HCl}$ , 8  $\text{H}_2\text{O}$ , 50  $\text{Et}_2\text{O}$ , 25  $\text{CHCl}_3$  and 2 of  $\text{NaOH}$  soln.; shake the mixt. If, however, 3 g. of the ext. are mixed with 57 g. of 1%  $\text{HCl}$ , and the soln. is filtered after 12 hrs., the result is much lower, due presumably to the pptn. of the acid-insol. alkaloid. ("No indication is given of the nature of the acid-insol. alkaloid, or whether it is actually present in the ppt."—Ed., *Pharm. J.*)

S. WALDBOTT

**The use of biological methods for the titration of certain toxic or medicinal substances.** A. RICHAUD. *J. pharm. chim.* 22, 257-65 (1920); cf. C. A. 8, 1188.—Such tests as the Focke-Joannin method for standardization of heart tonics are useful and at times indispensable; yet, biological differences in the test animals preclude an exact "titration" of the active principles, or even a strict comparison between 2 galenical preps. of the same kind. Besides, an observed effect might be due to a fraudulent mixt., e. g., of seabane and ouabain for the digitalis effect.

S. WALDBOTT

**Laboratory notes.** I. Detection of mercury in the presence of bismuth. C. H. BOLLINGER AND R. W. TERRY. *Midland Druggist* 54, 377 (1920).—In the usual qual. method, sep.  $\text{HgS}$  from  $\text{Bi}_2\text{S}_3$  by boiling with a 20%  $\text{HNO}_3$  which leaves  $\text{HgS}$  pptd. To identify  $\text{HgS}$ , dissolve it in  $\text{HCl} + \text{KClO}_3$  and convert into  $\text{HgI}_2$ . As the red color is often obscured by traces of Bi present, ext. the cooled  $\text{HgCl}_2$  soln. with  $\text{Et}_2\text{O}$  which leaves  $\text{BiCl}_3$  undissolved. The Hg in the evapd.  $\text{Et}_2\text{O}$  ext. may be identified as  $\text{HgI}_2$  or with  $\text{SnCl}_2$ . Or, extn. with  $\text{Et}_2\text{O}$  may be applied to the original soln. in which Hg must be present as  $\text{HgCl}_2$ . II. Titration of milk of magnesia. *Ibid.* 54, 378 (1920).—In the place of  $\text{H}_2\text{SO}_4$  in the U. S. P. IX method, the use of oxalic acid is recommended. Boil the magma with an excess of  $\text{H}_2\text{C}_2\text{O}_4$  soln. and titrate back while hot, with phenolphthalein as indicator. Very sharp end-points are thus obtained. III. Determination of protein in wine, beef and iron, N. F. IV. *Ibid.* 54, 378 (1920).—In detg. N by the Kjeldahl-Gunning method, the  $\text{NH}_3$  introduced by the Fe and  $\text{NH}_4$  citrate and  $\text{NH}_4\text{OH}$  must be subtracted, amtg. to about 35% of the  $\text{NH}_3$  evolved. To det. its amt., distil 25 cc. of the prepn. with 2 g.  $\text{Na}_2\text{CO}_3$  into a known excess of standard acid and titrate back. IV. Standardization of volumetric solutions with cochineal and methyl red as indicators. *Ibid.* 54, 377 (1920).—These indicators, recommended by U. S. P. IX, cannot be used with the 2 U. S. P. titration standards  $\text{Na}_2\text{CO}_3$  and  $\text{KHC}_2\text{H}_3\text{O}_6$ . B. and T. propose these methods: (1) Prep. a standard  $\text{H}_2\text{SO}_4$  with  $\text{BaCl}_2$ . Standardize an alkali against it, using any indicator. (2) Add anhydrous  $\text{Na}_2\text{CO}_3$  to a measured excess of the acid to be standardized. Expel the  $\text{CO}_2$  by heat, cool under a soda-lime tube, and det. the excess of acid by carbonate-free alkali of previously detd. standard. Then dil. the acid soln. to standard strength.

S. WALDBOTT

FALCK, A.: Die Arzneibücher (Pharmakopöen), vergleichend besprochen mit einem Verzeichnis der Arzneibücher. Leipzig: J. A. Barth. 168 pp. M. 24. For review see *Pharm. Weekblad* 57, 1353(1920).

GREENISH, HENRY G.: A Text-book of Materia Medica. 3rd Ed. revized. London: J. and A. Churchill. 568 pp. 27s. net. For review see *Pharm. J.* 105, 351 (1920).

Mercury compounds of glucosides. M. HARTMANN. U. S. 1,354,105, Sept. 28. Hg compds. are formed of glucosides, most of which are easily sol. in  $H_2O$  with a neutral or acid reaction and may be used for injections with but slight toxicity and without producing cutaneous necroses. The compds. are white powders difficultly sol. in org. solvents. The following examples are given: (a) amygdalin 3 and Hg acetate 3 are heated with  $H_2O$  100 parts to about  $70^\circ$  for 1-2 hrs. and as soon as tests with soda lye do not further ppt. yellow Hg oxide the soln. is evapd. to dryness *in vacuo* and the residue is pulverized with small quantities of alc. and ether which converts it into a fine cryst. powder which is filtered off, washed with ether and dried in an exsiccator; (b) salicin 2.9 and Hg acetate 3 are heated with  $H_2O$  100 parts until soda lye no longer gives a ppt. of Hg oxide, which requires about an hr., the mass is then evapd. *in vacuo* and pulverized with ether. A cryst. powder sol. in  $H_2O$  is obtained, which is difficultly sol. in abs. alc., ether and  $CHCl_3$ . Hg is split off from it by  $H_2S$  or by strong acids but not by alkalis; (c) arbutin 27 and Hg acetate 30 are heated with 50% alc. 200 parts for 1-2 hrs. at  $70-80^\circ$  and when a ppt. of Hg oxide no longer is formed by soda lye the mass is evapd. *in vacuo* to dryness and pulverized with ether.

Dentifrice. M. L. RHEIN. U. S. reissue 14,961, Oct. 19. See original pat. 1,297,494; C. A. 13, 1620.

## 18—ACIDS, ALKALIES, SALTS AND SUNDRIES

T. LYNTON BRIGGS

The manufacture of white arsenic. E. C. WILLIAMS. *Eng. Mining J.* 110, 671-3(1920).—Expts. conducted for the production of a dense white product free from Sb and Pb are described. A small reverberatory furnace, fired with coke, Cottrell plant product and blast furnace flue dust, is connected with cooling chambers. Most of the trouble that may develop is due to faulty feeding. The farther the  $As_2O_3$  is carried the lighter it becomes in density. Change of draft influences the Sb content and other impurities. Sb may be reduced to 0.3%, and Pb to traces by resmelting. The product deposited in the first half of the chambers is heavily crystallized. Since the Cottrell plant product is very inflammable, it is necessary to make hoppers airtight. Basic principles of furnace design and operation are: draft controls, impurities and to a great extent density; deeper cooling chambers produce a denser, cleaner, and whiter product; the cooling chambers and the furnace should be in line so that right angle turns may be eliminated. This secures great furnace draft with a minimum chamber draft. The cooling chamber should gradually be enlarged as it leads from the furnace.  $H_2BO_3$ , in powd. form or satd. soln., is an effective remedy for *As fume burns*.

W. H. BOYNTON

Steam consumption in the formation of ammonia from calcium cyanamide. JULIUS BAUMANN. *Chem.-Ztg.* 44, 293-4(1920).—Using a single autoclave, 331 kg. steam were required for 100 kg. cyanamide. If a group of autoclaves is used, this figure is cut to 126 kg.

C. J. WEST

Sources of American potash. R. O. E. DAVIS. U. S. Dept. Agr. Dept., *Circ.* 61, 7 pp.(1919).

Source	Actual production of $K_2O$ in tons			Estimated capacity in tons
	1916	1917	1918	
<b>Natural brines:</b>				
Nebraska lakes.....		14,558	28,854	50,000
Searles Lake, etc.....	3,994	6,092	10,862	28,000
<b>By-product sources:</b>				
Cement mills.....		1,621	1,549	3,500
Blast furnaces.....		185	230	.....
Molasses waste.....	1,845	2,846	3,467	4,000
Beet sugar factory waste.....		369	1,213	3,000
<b>Miscellaneous sources:</b>				
Alunite.....	1,850	2,402	2,621	4,000
Kelp.....	1,556	3,572	4,804	5,500
Wood ashes.....	412	621	673	1,000
Others.....	63	305	289	1,000
<b>Total.....</b>	<b>9,720</b>	<b>32,571</b>	<b>54,562</b>	<b>100,000</b>

F. M. SCHERTZ

The channel process of making carbon black. R. O. NEAL. *Chem. Met. Eng.* 23, 729-34(1920).—The main difference between the channel, small rotating disk, large plate and roller or rotating cylinder systems is in the size and shape of surface upon which the C is collected and rate at which the moving devices actuate. Details of plant construction are discussed. The gas, from the wells, discharge from gasoline plant, or gas transportation mains is reduced in pressure by suitable regulators, passed through a gasometer tank bringing the pressure below 1 oz. (28.3 g.) per sq. in. (sq. cm.). The channels upon which the black deposits are mild steel and supported by trucks running on overhead rails. C collecting hoppers are built directly below the channels and spaced 4 ft. apart. Two classes of scrapers are used, *i. e.*, those operating continuously and those directly in contact with the channels while the tables are moving in one direction only. The latter gives a more uniform product. Galvanized sheet iron screw conveyors have a 1" shaft supported every 8 ft. by a spider bearing or cast iron hub. The overflow tee located at the discharge end of the conveyor consists of a short pipe connected at an angle of 30° and carries a sheet iron cap. A pipe, 5' long, and crimped at the end is connected at right angles to the conveyor below the tee. The burners are located between the hoppers and 3-4" below the channels. They have lava tips made of selected steatite which has been heated to about 1093°, which insures hardness and strength. The common types burn 4-14 cu. ft. gas/hr. Uniformity of flame and gas consumption are essential. Each unit is equipped with a 20-h.p. internal combustion engine, 2-cycle type. Grit, scale and hard particles are removed by the bolting machines. Means of storage and packing are described. App. is fully illustrated.

W. H. BOYNTON

The disk, plate and cylinder processes for the production of carbon black. R. O. NEAL. *Chem. Met. Eng.* 23, 785-9(1920); cf. preceding abstract.—Description with mech. details of disk, plate and cylinder processes. The driving mechanism and heat variations are also discussed. The quantity and yield by the disk process is approximately the same as that made by the channel process. The plate or "Cabot" process is featured by the very slowly moving parts and by the small power requirements. The black is removed from the plates by scrapers placed radially to the mast. In the roller process the scrapers are located at or near the direct top of the rollers and run longitudinally the length of the rollers, having direct contact and scraping

continuously. A removable sheet iron hood is provided with apertures for controlling the ventilation with the chamber. The grade of black in this process is detd. by the rate of speed of the cylinders. The gear difficulties of this industry are overcome by the use of pinions with facial dimensions 4-5 times that of the gears. A recent device consists of a bevel wheel driven with a spur gear. Maximum service is obtained by correct installation, proper maintenance, lubrication where possible, inspection, and ample strength, ductility and wearing surface, hardness in gears and pinions. Production by a thermal decomposition of natural gas is not commercially satisfactory. The channel, rotating disk and large plate methods give approximately the same yield of black per 1000 cu. ft. of gas. The product from the roller system is more valuable, and is utilized for specialized purposes. Advantages of the channel process are: almost complete plant construction from standard forms and the cheapest construction. The large plate method is advantageous for large production, but large special castings make its initial cost high. Although requiring special castings the rotating disk process shows great elasticity which permits repairs without elimination of an entire condensing building. The roller system produces the smallest yield, yet requires the largest depositing surface per pound of black and per 1000 cu. ft. of gas burned.

W. H. BOYNTON

**Graphite.** BENJAMIN L. MILLER. *Mineral Ind.* 28, 314-28(1919).—A review of the industry in the United States and foreign countries. A. B.

**Gypsum.** FRANK A. WILDER. *Mineral Ind.* 28, 320-36(1919).—Production and utilization are discussed. A. B.

**Magnesite.** SAMUEL H. DOLEBEAR. *Mineral Ind.* 28, 433-41(1919).—Production and trade in the United States and foreign countries are discussed, with bibliography. A. B.

**Mica.** J. VOLNEY LEWIS. *Mineral Ind.* 28, 461-6(1919).—A review, with statistics. A. B.

**Monazite.** ANON. *Mineral Ind.* 28, 476-8(1919).—Statistics of production and trade. A. B.

**Phosphate rock.** WM. H. WAGGAMAN. *Mineral Ind.* 28, 534-48(1919).—Production and technology are discussed, with bibliography. A. B.

**Potash.** FREDERICK W. BROWN. *Mineral Ind.* 28, 508-83(1919).—The sources and output of potash in the United States and other countries are discussed, with statistics. A. B.

**Selenium and tellurium.** ANON. *Mineral Ind.* 28, 624(1919).—Notes on production and use. A. B.

**Sodium salts.** CHESTER H. JONES. *Mineral Ind.* 28, 625-40(1919).—Statistics on production and trade in nitrate, salt, and soda ash are given. A. B.

**Sulfur, pyrite and sulfuric acid.** ANON. *Mineral Ind.* 28, 641-55(1919).—A review of production and trade, with statistics. A. B.

**Talc and soapstone.** FREDERICK B. PECK. *Mineral Ind.* 28, 656-63(1919).—Production and imports are given and the use discussed. A. B.

**Titanium and zirconium.** ANON. *Mineral Ind.* 28, 685-7(1919).—A review of the industries. A. B.

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Industrial readjustments of certain mineral industries affected by the war (RIDDELL, *et al.*) 13. Recovering potassium compounds in cement manufacture (U. S. pat. 1,354,727) 20.

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**Nitric acid.** K. B. QUINAN. U. S. 1,355,357, Oct. 12. See Brit. 131,029 (C. A. 14, 97).

**Hydrocyanic acid.** G. H. BUCHANAN. U. S. 1,355,384, Oct. 12. HCN is produced by mixing an emulsion with  $H_2O$  of an impure cyanide containing substantially 40%  $CaCl_2$  and 15% lime with a dil. soln. of  $H_2SO_4$  and heating to evolve the HCN.

**Hydrofluoric acid.** VERBİN CHEMISCHER FABRIKEN IN MANNHEIM. Brit. 147,711, July 8, 1920. HF is prepd. from  $H_2SO_4$ , preferably in the form of monohydrate, and a natural fluoride such as fluorspar by thoroughly mixing equiv. quantities of the two substances with a solid such as finely ground anhydrous  $CaSO_4$  and heating to 200–300°. By feeding the material into a muffle furnace of the Mannheim sulfate type which is provided with mechanical stirring and discharging devices, the process becomes continuous and the handling of the resulting  $CaSO_4$  charged with HF is avoided. The HF may be collected as such, or it may be absorbed by means of alkalies, thereby yielding alkali fluorides. The residual  $CaSO_4$  is used in the prepn. of fresh charging material.

**Hydrochloric acid and alkali aluminium silicate.** A. H. COWLES. Ger. 318,030, Sept. 10, 1912. An intimate mixt. of clay, alkali chlorides and materials containing C (charcoal, hard coal, bituminous shale, sawdust, tartaric acid or  $Na_2CO_3$ ) is formed into sticks or briquets and dried and heated to a high temp. in a canal oven in an atm. containing water vapor. The temp. of the oven may vary between 900 and 1500°; the higher temp. being suitable to a higher content of clay in the mixt. The constituent of the mixt. containing C renders the mass porous as a result of its combustion, so that a large active surface is formed. The mixt. may contain 30–65% clay (inclusive of the chem. combined  $H_2O$ ) and 30–50% alkali chloride. The C content may be 3–10%.

**Acid liquor from niter cake.** W. C. HOLMES. U. S. 1,354,649, Oct. 5. Niter cake is treated with  $H_2O$  in insufficient amt. to dissolve it, until a liquor of an acidity of 20–30% is obtained. Counter-current extrn. is employed.

**Ammonia from cyanide furnace products.** F. J. MERTZGER. U. S. 1,354,574, Oct. 5.  $NH_3$  is produced without material loss from formation of ferrocyanide by treating a cyanide furnace product (such as formed from coke,  $Na_2CO_3$ , N and Fe at 1000°) with dry steam at a temp. above that of condensation but below 500°.

**Oxidizing ammonia.** C. L. PARSONS and L. C. JONES. Can. 205,204, Nov. 2, 1920. An app. for oxidizing  $NH_3$  has a contact agent in the form of a sheet so arranged as to have opposed heat-emitting surfaces.

**Titanium hydroxide.** F. E. BACHMAN. U. S. 1,354,040, Oct. 5. Ti hydroxide is recovered from solns. such as sulfate solns. containing Fe salts as impurities, by treating the materials, in dil. acid soln., with  $K_4Fe(CN)_6$  or  $K_3Fe(CN)_6$  to ppt. the impurities and then pptg. the Ti content of the soln. by diln. and boiling.

**Extracting alkali metals; carbon.** L. HACKSPILL and C. STAHLING. Brit. 148,122, July 9, 1920. Alkali metals are obtained by heating a halogen salt of the metal with an alk.-earth compd. such as a carbide, hydride, or silicate. E. g., a mixt. of NaCl and  $CaC_2$  may be heated in a vacuum first to 350° and afterwards to 800°, whereupon Na distils to the colder parts of the app. A high-quality lampblack is formed as a by-product.

**Alkali thiosulfate and carbonate from alkali sulfides.** A. CLEMM. Ger. 305,194, Nov. 18, 1917. The sulfide soln. is allowed to flow over finely divided, thoroughly ignited C offering a large surface, e. g., porous charcoal or a mixt. of similarly acting C substances, at the same time passing over the mass O or air and  $CO_2$ , whereupon the carbonate is removed from the soln. in the known manner.  $2Na_2S + CO_2 + 4O = Na_2S_2O_3 + Na_2CO_3$ . The sepn. of the thiosulfate and the carbonate can be effected readily by crystn. or salting out. In the prepn. of the K salts, the carbonate is sepd. in the form of the difficultly sol. bicarbonate.



Sodium cyanide. C. B. JACOBS. U. S. 1,354,561, Oct. 5. Coking coal and  $\text{Na}_2\text{CO}_3$  are intimately mixed and the mixt. is heated while in loose pulverulent condition to produce a porous coke containing  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{O}$  trapped within the cell walls of the coke. This product is treated with N while heated to produce NaCN.

Sodium cyanide. O. L. BARNESBY. U. S. 1,355,642, Oct. 12. NaCN is formed by heating activated C,  $\text{Na}_2\text{CO}_3$  and Fe or Fe ore in an atm. of N to about 800-1150°.  $\text{Ba}(\text{CN})_2$  may be similarly formed from BaO or  $\text{BaCO}_3$  at 1000-1500°.

Production of nitrates. JAS. R. PARTINGTON and ERIC K. RIDGAL. Can. 205,999, Nov. 30, 1920. App. is specified for the production of nitrates by the absorption of oxides of N. Cf. C. A. 14, 2399.

Aluminium chloride. F. C. FRARY. U. S. 1,354,818, Oct. 5.  $\text{AlCl}_3$  is made by adding Cl and a reducing agent such as coke to pulverulent Al dross. The reaction begins at 50° and is exothermic.

Potassium chloride from furnace dust. E. ANDERSON and F. S. MOON, JR. U. S. 1,354,842, Oct. 5. Furnace dust containing  $\text{K}_2\text{SO}_4$  and KCl is leached with a soln. containing sufficient  $\text{CaCl}_2$  to convert the alkali metal sulfates into chlorides and KCl is recovered from the soln. thus obtained.

Dehydrating calcium chloride and other substances. A. W. BROWNE. U. S. 1,354,279, Sept. 28.  $\text{CaCl}_2$  to be dehydrated is treated with a soln. of HCl in  $\text{CCl}_4$ ,  $\text{CHCl}_3$ , MeOH, EtOH, Et<sub>2</sub>O, acetone,  $\text{C}_6\text{H}_6$  or other non-aqueous solvent.

Manufacture of lead sulfate, sulfite and oxides from lead sulfide. J. GYTSHAM. Japan 35,836, Feb. 14, 1920. Volatilized substances, obtained by heating finely powdered PbS in an elec. furnace, are reacted on with O in hot state and the products are sepd. The operations are explained with diagrams.

Oxysalt compositions. CHAS. CATLETT. Can. 206,015, Nov. 30, 1920. A dry pulverulent material comprizing a set oxychloride compd. of an earth metal is made by causing an earth metal oxide and an earth metal chloride to react in the presence of moisture to form an oxychloride cement which is allowed to set and is then pulverized while still containing water of hydration. Cf. C. A. 13, 176.

Bismuth oxide. E. R. DARLING. U. S. 1,354,806, Oct. 5. Yellow Bi oxide is made by fusing metallic Bi with  $\text{AgNO}_3$  and passing Cl gas into the fused material.

Aluminium oxide. H. J. GOLDSCHMIDT. U. S. 1,354,824, Oct. 5. Plagioclases of the labradorite-anorthosite series or other materials containing Al and other metals such as Ca, K and Na are treated with  $\text{HNO}_3$  and the nitrates formed are subjected to a temp. of about 300° in order to convert the  $\text{Al}(\text{NO}_3)_3$  into oxide without affecting the other nitrates. Cf. C. A. 14, 1598.

Cuprous oxide. E. C. R. MARKS. Brit. 147,958, Feb. 1, 1918.  $\text{Cu}_2\text{O}$  is obtained by blowing air through liquid refined Cu until the desired stage of oxidation is effected. The cooled material may be ground; it is suitable for the production of Cu salts and cuprammonia solns.

Hydrogen and zinc oxide. R. H. MCKEE. U. S. 1,355,904, Oct. 19. Pure ZnO and H are prepd. by bringing substantially pure Zn in vapor phase into reactive contact with steam in sufficient excess to insure complete oxidation of the Zn.

Composition for liberating sulfur dioxide. L. G. WASSON. U. S. 1,356,029, Oct. 19. A dry, stable solid mixt. capable of evolving at least about 29.5% of  $\text{SO}_2$  upon treatment with  $\text{H}_2\text{O}$  is formed by mixing chem. combining proportions of  $\text{NaHSO}_4$  and  $\text{Na}_2\text{S}_2\text{O}_4$ . U. S. 1,356,030 relates to a compn. for the same purpose formed of  $\text{Na}_2\text{S}_2\text{O}_7$  and  $\text{Na}_2\text{S}_2\text{O}_8$ .

Separating bromine and chlorine. H. H. DOW. U. S. 1,354,472, Oct. 5. Gaseous mixts. (such as are obtained from brines by oxidation) containing both Br and Cl are treated with Fe and a soln. of an alkali halide containing some  $\text{Na}_2\text{CO}_3$  to effect selective absorption of the Cl.

**Regenerating mercury catalyzers.** ELECTRIC FURNACE PRODUCTS CO., LTD. Norw. 30,588, Apr. 6, 1920. The exhausted catalyzer is treated with Cl gas to convert the Hg into HgCl<sub>2</sub>, which is removed from the resulting mixt. and reconverted into the desired catalyzer.

**Oxidizing gases.** F. C. SCHMITZ. Can. 205,722, Nov. 16, 1920. Gases are oxidized by subjecting the flowing gas to the action of an opposing current of O. This method is specifically applied to the *manuf. of P<sub>2</sub>O<sub>5</sub>*.

**Gas for aerostats.** R. LE ROSSIGNOL. Brit. 148,008, May 7, 1919. Gas for aerostats comprizes a non-inflammable and non-explosive mixt. of H and He. The proportion of H preferred is  $\frac{1}{4}$  of the total vol.; the Provisional Specification states that this proportion may be as high as one quarter.

**Contact body with ceramic material as carrier.** V. ZIEREN. Ger. 317,979, Oct. 25, 1917. The contact body consists of a thin-walled porous member provided with channels to permit the passage of gas, and in certain circumstances with projections. The walls are covered completely with contact substance. The thickness of the walls does not exceed 2 mm., and the cross-section is not more than 20 mm.

**Soluble compounds from feldspar.** H. S. BLACKMORE. U. S. 1,355,381, Oct. 12. Finely divided orthoclase or a similar material is heated under pressure with a soln. of Al silicofluoride for 3-5 hrs. to form K<sub>2</sub>SiF<sub>6</sub>. Insol. matter is sepd. and the soln. is allowed to cool to effect deposition of K<sub>2</sub>SiF<sub>6</sub>. The aluminous residue obtained as a by-product may be employed as a china clay for manuf. of crockery. Na<sub>2</sub>SiF<sub>6</sub> and (NH<sub>4</sub>)<sub>2</sub>SiF<sub>6</sub> also may be used instead of (Al)<sub>2</sub>(SiF<sub>6</sub>)<sub>3</sub>. This method avoids the disadvantage of corrosive action of HF on the app. such as may occur when H<sub>2</sub>SiF<sub>6</sub> is used to decompose silicates. U. S. 1,355,588 relates to a similar method in which FeSiF<sub>6</sub> is used as the decomposing agent with or without the addition of (NH<sub>4</sub>)<sub>2</sub>SiF<sub>6</sub>, in obtaining sol. compds. from orthoclase, leucite, glauconite or similar minerals. The reaction is preferably carried out under 200 lbs. pressure per sq. in. at a temp. of about 175°.

**Phenolic condensation products.** L. H. BAEKKELAND. U. S. 1,354,154, Sept. 28. "Hexatriphenol" (hexamethylenetetraminetriphenol) or equiv. methyleneaminophenols derived from the cresols or phenolic mixts., containing either more or less than 3 mol. proportions of the phenolic compd. for each mol. proportion of (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub>, may be used to supply part or all of the CH<sub>2</sub> groups which are necessary to convert phenolic resins into potentially reactive substances or mixts. available for manuf. of molding products, varnishes and other purposes. The methyleneaminophenol serves not only as a conversion medium but as a good solvent for the phenolic resin and improves the workability of the material in the final manuf. It may also be advantageous to use such added solvents as C<sub>10</sub>H<sub>8</sub> or chlorinated C<sub>10</sub>H<sub>8</sub> derivs., phenolic esters, or other vol. or non-volatile solvents.

**Plastic compositions.** H. FELDMMANN. Brit. 148,117, July 9, 1920. A "spaddle mass" is made from a mixt. of celluloid soln. with white lead, "spaddle base," coloring matter or the like. In an example  $\frac{1}{4}$  kg. of celluloid waste is dissolved in 1 kg. of ethyl lactate and 5 l. of benzene are gradually added. To this soln. are added 5 kg. of white lead and 10 kg. of "spaddle base." Oil or fat may also be added. Amyl or ethyl acetate, benzene, methylated spirit, etc., may be used for dissolving the celluloid. The product may be applied to any base and smoothed by a single dry grinding.

**Plastic compositions.** GES. FÜR TORF-ISOLATION G. HUHN. Brit. 148,190, July 9, 1920. In the manuf. of insulation materials from a mixt. of peat and fusible binding agents, the mixt. is made in the cold and is subsequently heated and either subsequently or simultaneously pressed. As the temp. rises the pressure may be increased. Suitable binding agents are pitch, tar, resin, asphalt, etc.

**Adhesives.** L. KNORR. Brit. 148,216, July 9, 1920. A dry mixt. which produces an adhesive when stirred up with cold  $H_2O$  is composed of (1) a protein substance capable of forming salts, *e. g.*, casein, blood albumin, gluten, or albumoses, (2) a hydroxide of an alkaline earth, and (3) an alkaline salt of an acid which yields insol. or difficultly sol. alk.-earth salts, *e. g.*, alk. phosphates, carbonates, oxalates, silicates or fluorides. By the addition of  $NaCl$ ,  $CaCl_2$ , etc., the solution of the mixt. can be accelerated; or by means of  $BaCl_2$ , it can be retarded. Substances such as  $BaSO_4$ , gypsum, or chalk, may be added to alter the time of drying of the adhesive.

**Adhesives.** L. KNORR. Brit. 148,217, July 9, 1920. The compn. of the adhesive described in 148,216 (above) is modified by the whole or partial replacement of the alk.-earth hydroxide by the oxide or hydroxide of  $Mg$ .

**Composition for treating fibrous brake linings.** W. D. PARDOE. U. S. 1,354,996, Oct. 5. A compn. for treating fibrous material for the manuf. of brake linings is formed of asphalt 26 oz., a metallic oxide such as  $Pb_2O_3$ , 4 oz.,  $S$  5 oz., a vulcanization accelerator such as  $(CH_3)_4N_4$  and gasoline and  $C_6H_6$  or other volatile solvents.

**Coating containers for alkalis.** G. W. HOWLETT. U. S. 1,355,976, Oct. 19. Receptacles formed of paper or similar material and adapted for holding alkalis are coated with a soln. of cellulose nitrate or cellulose acetate.

**Electric insulating compositions.** BLERIOT, LTD. Brit. 147,746, July 8, 1920. A compn. for elec. insulation, especially designed for use with elec. heating and cooking app., consists of carborundum with a soln. of  $K_2SiO_3$  or  $Na_2SiO_3$  as a binding medium, with or without the addition of washed sand and powdered quartz or glass. The mixt. is baked at a temp. of 400–500°.

**"Turkish birdlime."** C. A. CLEGHORN. U. S. 1,356,267, Oct. 19. A working form of "Turkish birdlime" is formed by heating the material to about 80° and gradually stirring alc. into it. Borax, shellac and diatomaceous earth may also be added.

**Briquets.** O. DOBBELSTEIN. Brit. 148,115, July 9, 1920. Material to be briquetted, *e. g.*, non-bituminous coal dust, ore powder, etc., is mixed with powdered gas-coal dust and the mixt. is subjected to a pressure of 4,000–5,000 atms.

**Disinfectant and cleansing compositions.** A. PHILIPSON. Brit. 147,861, July 9, 1920. A solidified sol. disinfectant and cleansing compn. which dissolves readily in  $H_2O$  to produce a stable emulsion is prepd. from a tar oil, a fat, and alkali sufficient to neutralize the free acids of the tar oil and to saponify or partly saponify the fat. A soln. of a sol. disinfecting salt, such as oxalate of  $Na$  or  $K$ , may be added. Seven parts of tar oil are boiled with three parts of a soln. of  $NaOH$  of about 40° Bé. One part of animal or vegetable fat, such as coconut oil or stearic acid, is added for every 3 parts of the mixt. and boiling and stirring are continued, additional alkali, preferably  $NaOH$  or soda ash, being added in sufficient quantity to saponify or partly saponify the fat. The compn., with or without the addition of  $Na_2C_2O_4$ , etc., is poured into molds and sets to a stiff jelly. The proportion of alkali used is preferably such as to produce a substantially neutral product, and the quantity of  $H_2O$  used is such that thorough saponification is obtained before the excess not required in the final product has been lost by evapn. The compd. is stated to dissolve in  $H_2O$  with rapid disintegration or effervescence.

## 19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

G. E. BARTON, A. V. BLEININGER

**The melting of colored glasses.** ANON. *Glasindustrie* 31, 209–10(1920).—An alk. mix is the usual basis of colored glasses. The yellows are very difficult to pro-

duce. Twenty formulas for yellow glass are given, 10 for blue, 32 for green, 2 for gold ruby, 2 for copper ruby, 2 for gray, 2 for black and 2 for brown. C. H. KERR

Absorption of heat in glass. A. Q. TOOL AND C. G. EICHLIN. *J. Optical Soc. Am.* 4, 340-63(1920); cf. *C. A.* 14, 2245.—A marked increase in the absorption of heat by glass heated at a uniform rate takes place within a narrow temp. range slightly above the annealing temp. The authors suggest as the cause the formation of one or more types of mol. aggregates possibly of cryst. structure. Tests on chilled and annealed glass produce the results expected from this theory and also lead to the possibility, "that annealing is not alone a question of removing stresses, but also of producing homogeneity." The results of various tests on 20 types of glass are tabulated.

DONALD E. SHARP

Cause of blisters in glassware. T. D. LONG. *Glassworker* 39, No. 52, 18(1920).—Blisters are caused by vacuum due to cooling too long. An expt. is described to prove this. A gathered piece held in a stream of compressed air will form blisters on the inside. On heating in a glory hole they will disappear and will not reform on cooling if the cooling is properly done.

R. J. MONTGOMERY

Glass container research work. J. M. HAMMER. *Glassworker* 40, No. 1, 11 (1920).—A general description of the work being done by A. W. Bitting in the lab. of the Glass Container Assoc. of America.

R. J. MONTGOMERY

Note on the tensile strength of refractory materials. MORRIS W. TRAVERS. *J. Soc. Glass Technology* 4, 138-9(1920).—In many places in furnace construction and especially in pot walls, tension and not compression is the important property. Changes which increase resistance to load may have a very different effect on tensile strength. The theory that an ideal refractory should consist of a very high percentage of infusible material, carefully graded and bonded with a very small quantity of vitreous bond may be justified from the standpoint of resistance to load, but it does not follow that tensile stresses will be equally well withstood. The ideal refractory consists of a system of interlocking crystals formed *in situ*, in a glassy matrix. If the matrix is such as to form a sillimanite layer in contact with alkaline fluxes, then corrosion by glass is largely avoided, for such a layer greatly delays the mixing of the liquid phases on either side of it.

C. H. KERR

Constitution of silicates (JAKOB) 6. Study of transition points by a dilatometric method (BRAESCO) 2. Composition of Post-Tertiary clay material (TAMM) 8.

SPRINGER, LUDWIG: *Laboratoriumsbuch für die Glasindustrie*. Verlag Halle, Wilhelm Knapp. 142 pp. M. 12, bound M. 14, 80 + 30%. Verlagszuschlag. For review see *Z. angew. Chem.* 33, II, 392(1920).

Abrasive mixture. B. E. WISE. U. S. 1,353,979, Sept. 28. Mixts. for use on razor strops are formed of powdered carborundum 4-8, powdered aloxite 0-4, powdered talc 2, petrolatum 0-8 and paraffin 0-5 parts.

Refractory furnace lining. A. F. QUIN and C. LACEY. U. S. 1,355,689, Oct. 12. A compn. formed of mica 6, asbestos 8 and salt 2 parts, ground and mixed, is used for making bricks for lining furnaces.

Refractory support for ceramic ware during firing. S. C. LINBARGER. U. S. 1,356,211, Oct. 19. A support for ceramic ware which does not cause its discoloration during firing is made of Si carbide with a glaze coating formed of flint 1100, clay 800, feldspar 10 and whiting 10 parts.

## 20—CEMENT AND OTHER BUILDING MATERIALS

C. N. WILEY

Thermal phenomena in the setting of portland cement. F. KILLIG. *Concrete* (Mill Sec.) 17, 120(1920).—See C. A. 14, 3515. J. A. MONTGOMERY

Corrosion tests. ANON. *Concrete* (Mill Sec.) 17, 126(1920).—An investigation carried on by the National Lime Association gave results which indicate that lime can be used to advantage in gypsum construction for the purpose of preventing the corrosion of reinforcing steel. J. A. MONTGOMERY

Action of alkali soils and waters on concrete. A. S. DAWSON. *J. Eng. Inst. Canada* 3, 476-80(1920); *Eng. Cont.* 54, 388-9(1920).—Soap and  $\text{Al}_2(\text{SO}_4)_3$  afforded some protection to concrete against alkali; however, soap reduced the tensile strength somewhat. The mixing of cement with weak solns. of  $\text{H}_2\text{SO}_4$ ,  $\text{Na}_2\text{HPO}_4$ ,  $\text{MgF}_2$ , and  $(\text{COOH})_2$  has been shown by lab. tests to increase the alkali-resisting qualities of concrete. According to D., the effects of most of the alkalies have also been shown to be less pronounced on neat cement briquets than on sand cement briquets, and in a degree proportional to the amt. of sand. J. A. MONTGOMERY

A contribution to the dispersoid and colloid chemistry of plaster. I. WO. OSRWALD AND P. WOLSKI. *Kolloid.-Z.* 27, 78-92(1920).—The older theory of the setting of plaster, which attributes the process to the interlocking of needle-like crystals of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  deposited from a supersatd. soln., cannot account fully for the facts. A number of investigators have suggested that colloidal phenomena play a part, but have not been able to study the question because of the lack of a suitable method for following the kinetics of setting experimentally. O. and W. find that the observation of the change in viscosity of dil. suspensions of plaster with time after prepg. affords the desired exptl. method. The detns. were made in an Ostwald type viscosimeter. The viscosity-time curves are S-shaped, the viscosity rising slowly at first, then more rapidly, and finally becoming const. at its max. value. The time required to reach the max. measures the time of setting of the plaster. The concn. of suspension suitable for these expts. lies between 2 and 5%. The higher the temp. the lower the max. viscosity. In one case the max. viscosity at  $0^\circ$  was 350 units, at  $18^\circ$ , 110 and at  $60^\circ$ , 23. Freshly ground plaster gives a higher viscosity than the same plaster after standing a few days. The dispersity of the plaster has an enormous influence, the finer the particles the higher the max. viscosity of the suspension. Added substances have an important influence. KCl causes the max. viscosity to be reached more quickly, but at a lower value.  $\text{NH}_4\text{Cl}$  gives a lower max. viscosity. With acetic acid the max. is reached more slowly, but is at least as high as the suspension with no addition. Further work is in progress. F. L. BROWNE

The manufacture of lime for chemical and metallurgical purposes. R. K. MEADE. I. *Chem. Met. Eng.* 23, 841-4(1920).—M. discusses different types of shaft kilns and describes an improved vertical type. There is a separate feed, and the burning is done between the ports of the furnace. Dimensions of the kiln are detd. by local conditions. Reinforced concrete shells are recommended as substitutes for steel. The draft and distribution of heat and fuels, their consumption and quantities of lime burned per ton are discussed. Producer-gas kilns are more economical in fuel and labor than grate-fired kilns. Points to guard against in the application to the vertical kilns are: proper mixing of the gas and air in the shaft, and preventing accumulation of lime in the gas ports. II. *Ibid* 873-5. A description of some applications of the rotary lime kiln in burning stone and recovering spent lime. This type yields a more evenly and more perfectly burned lime. Application is made in the burning of lime sludge from paper pulp mills, caustic soda works and beet sugar plants. The

sludge is largely de-watered by passage through a rotary drier. Lime recovered from the kiln consists of oval lumps from dust up to the size of a walnut. They slake and therefore causticize more slowly than rock lime. This pptd.  $\text{CaCO}_3$  settles more quickly. With careful handling the recovered lime will contain as much  $\text{CaO}$  as the best lump lime made from stone. The size and the hardness of the stone are of least importance in the rotary types, though it is best to screen out the dust from the coarse rock for the sake of purity of product.

W. H. BOYNTON  
Charring does not preserve wood. ANON. *Eng. Contr.* 54, 421(1920).

J. A. MONTGOMERY

TEDESCO, N. DE AND FORESTIER, V.: *Nouveau manuel théorique et pratique du constructeur en ciment armé*. 2nd Ed. Paris: Ch. Beranger. 316 pp. For review see *Chimie & industrie* 4, 572(1920).

Recovering potassium compounds in cement manufacture. C. CATLETT. U. S. 1,354,727, Oct. 5. Recovery of K compds. in treating a portland cement mixt. (which may contain feldspar to increase the K content) is facilitated by the addition to the mixt. of 1-5% of a set Ca oxychloride compn. of the sord cement type. Sufficient of the latter is used to supply Cl to combine with the K to be recovered. The volatilized K values leaving the kiln with the dust may be recovered by simple subsidence, use of wet or dry filters or by elec. pptn. The recovered material may be used as a fertilizer directly or may be leached for the recovery of K compds. in crystd. form. The use of Ca oxychloride is stated to have the advantage over NaCl that the fume and gases produced are much more readily recovered by elec. pptn. and no detrimental effect on the cement clinker is produced. Oxychlorides of Mg and Zn also may be used. The various oxychlorides (including a product formed by treating bittern with  $\text{Ca(OH)}_2$ ) may also be used for treating feldspar to produce sol. K compds., with or without simultaneous use of  $\text{CaCO}_3$ ,  $\text{CaSO}_4$  or other auxiliary substances.

Plastic coating material containing cement. J. C. DOBBINS. U. S. 1,355,131, Oct. 12. A coating material in paste form, adapted for use on walls or fabrics, is formed of portland cement 100, hydrated lime 13, a coloring material and mica 1.3 parts,  $\text{H}_2\text{O}$  and Na silicate soln.

Asbestos cement building material. R. V. MATTISON, JR. U. S. 1,355,406, Oct. 12. An asbestos cement suitable for building bricks, roofing or flue linings is formed of portland cement and finely ground serpentine. The serpentine used contains microscopic fibers of asbestos and a considerable proportion of ground quartz.

Preparing pumice stone for use in concrete. E. C. BAYER. U. S. 1,354,233, Sept. 28. See Brit. 135,476 (*C. A.* 14, 1025).

Preserving wood. F. BUN. Brit. 148,137, July 9, 1920.  $\text{HgCl}_2$  soln. is rendered more effective as a wood preservative by mixing with it a soln. of a fluorine salt such as NaF, a fluosilicate, or a mixt. of fluorine compds. with fluosilicates. The specification states that the  $\text{HgCl}_2$  is converted into complex salts. The salt solns. may be applied separately to the wood.

## 21—FUELS, GAS, TAR AND COKE

J. D. PENNOCK

The fossil fuels and their utilization during 1919. A. FURTH. *Z. angew. Chem.* 33, I, 205-8, 217-20, 223-5(1920); cf. *C. A.* 14, 818.—A very comprehensive review of the periodical literature and patent files, under the following headings: (1) Raw materials; (a) mining, (b) washing and dressing, (c) chem. compn., (d) fusibility of ash,

(e) proximate constituents, (f) utilization of low-grade fuels, (g) spontaneous ignition of coal, (h) briquetting, (i) powdered coal combustion, (j) chem. analysis, (k) calorimetry; (2) condensation, handling and purification of the gases; (a) new app., (b) elec. purification, (c)  $\text{NH}_3$  and S washing; (3) measuring, distribution and utilization of the gas; (a) metering, (b) gas burners and gas heating; (4) calorimetry and analysis of technical gases; (a) calorimetry and d. detn., (b) analytical methods, (c) S detn., (d) automatic analysis app., (e) limits of combustion of gas mixts.; (5) production, treatment and utilization of by-products, (a) ammonia, (b) benzene, (c) coal tar, (d) low-temp. tar, (e) naphthalene and (f) gas purification residues. W. B. V.

An inventory of our (German) fuels with respect to the newer processes of utilization. ED. GRAEFE. Dresden. *Brennstoff Chem.* 1, 6-8(1920).—The present estimates of the coal deposits in Germany fail to take any note of the important distinctions of quality between various deposits; the two handbooks which list the properties of German coals (Langbein and Schwackhöfer) give no data on the coking properties and yields of technically important by-products. The author calls attention to the necessity of a systematic research covering this field. W. B. V.

Prospects for supplying the German industries with coal, and means for the better utilization of fuels. DE GRAHL. *Z. Ver. Zuckerind.* 70, 357-67(1920).—An address. F. W. ZERBAN

The purpose and results of recent scientific investigations on coal. BÖRNSTEIN. *Z. Ver. Zuckerind.* 70, 369-81(1920).—A review, calling attention particularly to the results of recent work on the low temp. distn. of coal. F. W. ZERBAN

Coal saving in the chemical industry. DAVID BROWNLIE. *Chem. Trade J.* 67, 247-50, 311-4, 343-5(1920).—A report of conclusions based on personal inspection of 60 typical steam-boiler plants of chemical industries of Great Britain. The av. net efficiency of the boiler plants in question is only 58%; by reorganizing them along modern scientific lines it may be raised to 75% with a consequent saving of 23% in the coal bill. Faults pointed out are (1) improper design of grates; (2) ignorance of heating value of coal used; (3) lack of records of water evapn.; (4) improper treatment of feed water, with resultant scale troubles; (5) failure to use economizers, CO<sub>2</sub> recorders and superheaters. In addition chimney drafts and steam jets are used where mechanical draft should be employed, brick work and pipe covering are defective and firing is poor. As to the cause, "the chief engineer of many chemical plants is merely a glorified mechanic without scientific experience or education, paid and treated accordingly." H. L. OLIN

Conservation of coal. THOS. GOULDEN. *Gas J.* 152, 200-1(1920). J. L. WILEY

Thornley coalometer. R. P. NICHOLS. *Gas Age* 46, 379-80(1920); *Iron Age* 106, 588(1920); *Coal Age* 18, 485-6(1920).—A device is described for indicating temps. of soft coal while in storage. It consists of a set of temp. indicators encased in a long pointed steel tube, carrying at varying depths metal bulbs, and at its upper end a set of dials and pointers, which indicate, under all atmospheric conditions, the exact temp. of the coal in the vicinity of the bulbs. A spacing of 50 ft. between instruments is sufficient for efficient indications. J. L. WILEY

Bituminous storage practice. H. H. STOEK, C. W. HIPPAARD AND W. D. LANGTRY. *Univ. Ill. Bull.* 17, No. 21, 157 pp. (1920).—An exhaustive treatment of the subject based on data obtained from (1) a questionnaire sent to 200 railroads, coke plants, etc., which had stored coal under widely differing conditions; (2) a similar questionnaire returned by 300 power plants in Illinois; (3) a study of fires in coal piles in Chicago and in other cities; and (4) various authorities in this field. H. L. OLIN

Oxidation of coal. MARCEL GODCHOT. *Bull. soc. chim.* 27, 735-7(1920).—See C. A. 14, 3306. E. H.

The recognition of substances similar to coal. FRITZ HOFFMANN. Breslau. *Brennstoff Chem.* 1, 2-3(1920).—The tarry products obtained when sodium phenolate is heated for some time in an inert atm. at 485-490° were isolated and studied. The tar is pptd. on acidifying the water soln. of the melt; the Et<sub>2</sub>O-insol. portion is extd. with acetone and the soln. (A) reprecipitated with Et<sub>2</sub>O or benzene. Analysis of the brownish black powder which results, leads to the empirical formula (C<sub>7</sub>H<sub>5</sub>O)<sub>x</sub>. Mol. wt. could not be detd. because all of the solns., including that in dil. NaOH, proved to be colloidal; but it is suggested that *x* may be 6, and the structure that of hexa (hydroxyphenyl) benzene, being formed by the condensation of 7 mols. of PhOH with loss of H<sub>2</sub> and H<sub>2</sub>O. No derivs. were prepd. on account of the colloidal nature of the solns., but on destructive distn. of the dry powder, a small amt. of red-brown tar was formed and a coke remained which was insol. in NaOH, C<sub>6</sub>H<sub>6</sub>N and acetone. The residue from the acetone extn. of (A) was sol. in C<sub>6</sub>H<sub>6</sub>N and NaOH, and contained by analysis, ash 5.5%, C 72.8%, H 4.3%. W. B. V.

Coal as a future source of oil fuel supply. ARTHUR DUCKHAM. *Gas World* 73, 332-3(1920); *Chem. News* 121, 223-6, 237-8(1920); *Gas J.* 152, 217(1920).—A paper read before the Inst. of Petroleum Technologists at the Royal Soc. of Arts. D. believes that the industrial future of the country lies in the conversion of coal at the mouth of the mine into liquid and gaseous fuels and their transport through pipe lines to the consumer. He discusses the various processes of coal carbonization, saying that in a few years coke ovens will be obsolete as gas instead of coke will be the chief metallurgical fuel. The ideal system would be the complete gasification of coal, preferably by the single stage process, with the recovery of the max. amt. of gas and liquid fuel with a minimum of "cracking." The gas should be stripped of all satd. hydrocarbons and the max. amt. of N recovered. The plant should be designed to work with any coal and be composed of one or more vertical retorts superimposed on a water-gas producer, the coal being fully converted into coke before entering the producer and the latter being so built as to obviate the formation of hard clinker. Such a plant, fitted with recuperators and waste-heat boilers, would have an efficient heat cycle, and would conserve a large amt. of fuel. The gas could be delivered to consumers at from 1½ to twice the amt. it would cost per heat unit to supply solid fuel. The saving in labor and freight charges, and the increase in efficiency of utilization would more than compensate for the increased charge. Also in *Pet. Times* 4, 429-32(1920). J. L. WILBY

The transition to lignite firing in starch factories and drying plants. HERMANN GESELL. *Z. Spiritusind.* 43, 267-8, 279(1920); cf. *C. A.* 14, 3308.—The increased grate surface necessary to obtain undiminished evapn. from boilers transferred to lignite firing may often be obtained by using boilers normally held in reserve for periods of peak production. Water-tube boilers can usually be reset so as to increase the grate surface markedly, and so permit full evapn., provided the draft is high enough. Plants manufacturing moist starch can buy elec. power more cheaply than they can run small steam engines. Coke can hardly be replaced in the drum dryers, because the products of combustion come in contact with the moist material. Potato-drying plants can be operated best in connection with a distillery, since high-pressure steam is necessary. On account of the three-fold weight of fuel which must be handled, proximity to the mines attains much greater importance than heretofore. W. B. V.

Recent developments in furnaces for solid fuels. PRADEL. *Feuerungstechnik* 9, No. 2, 13-17(Oct., 1920).—Review of German patents, with 10 figures.

Gas producers. K. BUNTE. *J. Gasbel.* 63, 541-5(1920).—B. explains the advantages to be derived from heating gas retorts from central gas-producer plants and traces the development of gas-producer construction and operation from the original



step-grate type to the modern revolving-grate type with by-product recovery. The utilization of inferior fuels is also considered. J. L. WILEY

Gasification of coal in rotary ovens. ANON. *J. Gasbel.* 63, 567-8(1920).—Distn. of coal is effected in rotary kilns like those used in making cement. From 1 ton of run-of-mine gas coal were obtained 100 kg. water-free tar, 30 kg. light oils, 150 cu. m. of gas of 7000 cal., and 650 kg. of coke after deducting the amt. necessary to heat the ovens. The gas had an av. % compn. of  $\text{CO}_2$  6.3,  $\text{C}_2\text{H}_4$  10.6,  $\text{O}_2$  2.0,  $\text{CO}$  4.6,  $\text{H}_2$  0.0,  $\text{CH}_4$  64.6, and  $\text{N}_2$  11.9. 90% of the heat value of the coal was recovered. The coke can be further gasified in producers or be efficiently burned under boilers. It also briquets well, requiring less pitch for binder than coal briquets. J. L. WILEY

Heating the chamber ovens of the Kiel Gas Works with central generators. K. BUNTE, A. VIERHOFF AND E. CZAKO. *J. Gasbel.* 63, 589-94(1920); cf. Voigt, *C. A.* 14, 2407 and Friese, 3778. Results are given in detail of a 5-day test made on heating recuperatively a block of 26 Otto ovens with gas from 2 Otto gas generators. The thermal efficiency of the water-gas process was 78% with an added 6-7% for steam and hot water production. The loss through unburned fuel was only 1-2%. Nut coke was used. The generator gas had an av. % compn. of  $\text{CO}_2$  4.93,  $\text{O}_2$  0.12,  $\text{CO}$  27.97,  $\text{H}_2$  10.9,  $\text{CH}_4$  0.35,  $\text{N}_2$  55.72, calorific value 1185. The ovens were charged with a coal containing 14% ash and 28.9% volatile. The gas yield was 30.05 cu. m. per 100 kg. of coal, heating value 4880 al., compn.  $\text{CO}_2$  4%,  $\text{C}_2\text{H}_4$  2.4,  $\text{O}_2$  0.5,  $\text{CO}$  7.3,  $\text{H}_2$  42.9,  $\text{CH}_4$  25,  $\text{N}_2$  17.9, coke yield 68.43%. The fuel consumption for heating was 11.6 kg. per 100 kg. of coal coked or 38.8 kg. per 100 cu. m. of gas produced. J. L. WILEY

Observations on the production of generator gas from bagasse at the Technical Bureau Soenda. W. A. HATTINK. *Arch. Suikerind.* 28, 1539-76(1920).—This is a continuation of the article previously noted (*C. A.* 14, 3519), giving in great detail the figures on which the tables and graphs in the former paper were based.

F. W. ZERBAN

Low-grade gas-carbonizing methods. JAMES JAMIESON. *Gas World* 73, 345-9 (1920); *Gas J.* 152, 334-7(1920).—A low-grade gas of 400 B. t. u. or lower is being distributed in Greenock with excellent results for incandescent lighting, street lighting, and industrial uses such as furnace and rivet heating and core drying. Delivery of the extra vol. of gas required is accomplished by increasing the pressure, and admitting the high-pressure gas into each low-pressure district through suitable regulators. At present 25% of the max. load is being delivered at a cost of 0.49 d. per 1000 cu. ft. The vertical retort and water-gas process for the manuf. of a low-grade gas is no doubt the most efficient, according to the results obtained at Uddingston (*C. A.* 14, 2409), where one coal of const. compn. is used. But under the varying conditions met in small works and where several coals have to be used the production of water gas of const. compn. in vertical retorts is a doubtful proposition. It is also necessary to devise means of controlling absolutely the vol. and temp. of the steam entering the retorts. A flexible system of retort heating is important also. For the production of good water gas high temp. is necessary. The application of external heat is not the most efficient method but is at present the only practicable and profitable one. Internal heating by means of steam highly superheated by the waste gases would be ideal and very efficient but probably not economically obtainable, while superheating in a separately fired superheater would be financially prohibitive. Moreover, the destructive effects of superheated steam on pipes, valves, brick work, etc., are well known. Work has been done also on producing water gas in separate producers and passing it through the vertical retort either alone or with steam. A K. and A. plant was used, description of which is given. From each ton of mixed coal and coke (33:67), 57,000 cu. ft. of gas were obtained, after allowing for fuel to the boilers, of 325 B. t. u. with a high content of H and

CO and a low content of inerts. Rather favorable results were obtained also from this app. in exptl. work on complete gasification. The method of external heating of the entering fuel and its ultimate gasification by highly superheated gas being taken advantage of.

J. L. WILEY

**Problems of complete purification.** ANON. *Times Eng. Supp.; Gas J.* 152, 143 (1920).—Two processes of recent origin for effecting purification of gas from S by catalytic means are of interest. The Carpenter-Evans process accomplishes the removal of about 80% of the impurities by causing the  $CS_2$  to react with H, employing a catalyst of Ni reduced from the chloride. The disadvantage is that it is intermittent in operation due to the choking of the catalyst by the solid C residue left from the conversion of  $CS_2$  to  $H_2S$ . This deposit needs to be burned off at intervals. The Rideal-Taylor process is continuous in operation, the reaction being carried out with the aid of water vapor and a catalyst of  $Fe_2O_3$  activated by the addition of  $Cr_2O_3$ . The C in this case passes off in the form of  $CO_2$ . At a temp. of  $350^\circ$  over 90% of the S impurities may be removed by the addition of 7% by vol. of steam passed in with the crude coal gas at the inlet of the preheater immediately before the catalyst vessel. The temp. of satn. with water vapor and the temp. of the catalyst depend on the compn. of the gas, its S content and the nature of the catalyst employed. With systems of hot purification, it seems probable that COS is produced when a gas containing  $H_2S$  and CO is treated catalytically. Therefore the treatment cannot be applied advantageously to crude gas. In developing a practical process for oxidizing both  $H_2S$  and S compds. at high velocity to S or  $SO_2$ , 3 points are important: The choice of a suitably activated catalyst unaffected by poisoning influences such as cyanides, the provision of an absolutely tar-free gas, and an economical source of heat for carrying on the reaction.

J. L. WILEY

**Alkalinity tests in purification.** C. H. STONE. *Am. Gas Assoc.; Gas Age* 46, 299-301 (1920).—It is found that alkalis increase the efficiency of many oxides and that temp. and moisture of the oxide deserve more extended study. There appears to be no feasible way of setting a definite figure as to the best moisture content for all oxides. The efficiency of many oxides can be increased by making the boxes alkaline, the addition of 2% of lime giving the best results. Fresh lime should be added occasionally, as it is used up in the purification, one test showing a drop from 5.1 to 3.6% after the first fouling and to 2.6% after 3 foulings. Alkalinity did not apparently prevent the formation of  $FeS_2$ ,  $FeS$  or  $FeSO_4$ . The alkali added apparently accomplished its purpose by uniting with acids formed in the purifiers such as  $H_2SO_4$  and HSCN. Temp. expts., while not complete, show a relation between  $H_2S$  absorption and temp.: up to  $110^\circ$  F. as the temp. is increased the % of  $H_2S$  removed increases rapidly, while above  $110^\circ$  F. the velocity of the reaction becomes so great that S compds. are evolved. As recent work shows that there is but one hydrate of  $Fe_2O_3$ , the question of increasing the water of hydration has been eliminated, and future expts. will be made in d t g. the effect of hydration on the efficiency of an oxide for purification purposes.

J. L. WILEY

**New purifying plant at the Grasbrook Gas Works, Hamburg.** KRAUSE. *J. Gasbel.* 63, 611-5 (1920).—Description and operating details are given of a purifier installation designed to purify 720,000 cu. m. of gas per day. The purifying material is 90% "Lux" and 10% natural ore. Its period of activity was  $10\frac{1}{2}$  months with a final S content of 48.7%.

J. L. WILEY

**Ensuring a constant quality of water gas.** New automatic controller. ANON. *Gas World* 73, 297-8 (1920).—A device is described which automatically controls the flow of steam to the generator according to the fall of temp. in the fuel bed during the run. A slight reduction in gas capacity results, practically accounted for by the les-

sened amt. of  $\text{CO}_2$ , the loss in thermal output amounting to only from 0.15 to 0.8%. A saving of upwards of 11% is effected in steam consumption. J. L. WILEY

Chemical control of gas operation. F. J. KENNEDY. *Gas Age* 46, 342-5(1920).—Review of some of the important tests required for the chem. control of gas works. J. L. WILEY

Making and use of blue water gas. FRED H. WAGNER. *Gas Age* 46, 336-9 (1920).—An illus. description of the blue water gas plant at the Elrama Works of the Equitable Gas Co., Pittsburgh. J. L. WILEY

Technical and experimental tests on heating inclined chamber ovens at the Pasing Gas Works. E. TERRES, M. WALTER AND K. BRÜCKNER. *J. Gasbel.* 63, 605-11 (1920).—Operating details and data are given for an inclined chamber oven installation heated with generator gas. Special study was made of combustion in the flues, the effect of gas compn. on the thermal efficiency, and analytical and thermal examn. of the generator gases, with the idea of furnishing some logical basis for operation and construction. J. L. WILEY

Industrial heating with town gas in Birmingham. C. M. WALTER. *Gas J.* 152, 208-9(1920); *Gas World* 73, 363-5(1920). J. L. WILEY

Industrial use of town's gas in Sheffield. S. T. S. MUSGROVE. *Gas J.* 152, 207-8(1920). J. L. WILEY

French experience with gas-fired boiler installation demonstrates possibility of complete combustion of low B. t. u. gas without preheating supporting air. JOHN H. BARTLEY, JR. *Am. Gas Eng. J.* 113, 321-4(1920); *Iron Age* 106, 1261-2 (1920); *Chem. Met. Eng.* 23, 1033-5(1920).—A description is given of tests recently made on firing a water-tube boiler with lean producer gas at Montrambert (France) with the aim of utilizing mine slack and low-grade coals containing 42-45% of a somewhat fusible ash and 18-20% of volatile matter. The coal is first coked in coke ovens, the by-products being recuperated and the waste heat employed for steam generation, the coke utilized in gas producers and the surplus gas employed in internal combustion engines for generation of electricity. The gas is practically const. in compn. and calorific value. The boilers are fired by the surface combustion method. About 70% of the N in the coal is recovered as  $\text{NH}_3$ . The av. production of steam per boiler was 4200 lbs. per hr. with a gas consumption (corrected) of 69,600 cu. ft. The calorific value of the gas was from 91.3 to 101 B. t. u. per cu. ft. The boiler efficiency was 73.3%. Cf. Blache, *C. A.* 14, 461. J. L. WILEY

Smoke problem—coal fires vs. gas fires for domestic heating. J. H. BREARLEY. *Gas World* 73, 269-71(1920); cf. Fishenden, *C. A.* 14, 3146.—The position as to fuel economy seems to leave little to choose between domestic gas heating and heating by coal as it exists to-day. But the prospects for future ultimate economy certainly favor gas. J. L. WILEY

Notes on the gas regulation act. T. P. RIDLEY. *Gas J.* 152, 84-7(1920); *Gas World* 73, 278-8(1920); cf. *C. A.* 14, 3148. J. L. WILEY

Practical application of the gas regulation act, 1920. N. H. HUMPHRYS. *Gas World* 73, 263-6(1920).—Calorimetric phases are discussed (cf. *C. A.* 14, 3148). J. L. WILEY

New patents on the preparation of generator gas. GWOSDZ. *Feuerungstechnik* 8, 173-6(1920).—Review. C. J. WEST

Inflammability and explosibility of benzine and benzene. WA. OSTWALD. *Feuerungstechnik* 8, 191-2(1920).—A review with diagrams, which shows the known fact that benzine is more inflammable than benzene. C. J. WEST

What is the source of the benzene in coke ovens and gas retorts? FRANZ FISCHER AND HANS SCHRADER. *Brennstoff Chem.* 1, 4-6(1920); cf. *C. A.* 6, 2618; 11, 1036; 13,

178.—Low-temp. tar consists essentially of hydrocarbons similar to those of petroleum, and of phenols, but no aromatic hydrocarbons. The benzene homologs in coal tar must be formed from the phenols, since the other group can only be converted into aromatics to the extent of a few %. In support of this view the authors report the result of passing mixts. of  $H_2$  with phenols and hydrocarbons through tubes heated to  $750^\circ$ . Sepn. of free C was found to depend largely on the nature of the tube wall, being negligible in a porcelain tube and very considerable in an Fe tube; tinned iron was found to be a technically practical material which did not cause C sepn. Under these conditions, the phenols were reduced to benzene homologs, and the latter partly to benzene, while hexane was nearly unchanged, as shown by the following table:

	Yield of aromatic hydrocarbons, %	
	Tinned tube	Iron tube
Three mixed cresols.....	64	5
o-Cresol.....	72	35
1,3,4-Xylenol.....	72	5
Hexane.....	7	4
Solvent naphtha I.....	61	63
Solvent naphtha II.....	49	48

It is suggested that this process might be used to reduce the phenols of low-temp. tar to aromatic hydrocarbons, using coke-oven gas as the reducing agent. W. B. V.

Determining the influence of excess gas on fuel consumption in a gas-fired oven furnace. O. LELLEP. *Am. Gas Assoc.; Am. Gas Eng. J.* 113, 397-8(1920).—It was established by expts. that the most economical way of heating a furnace is to use a correctly proportional mixt. of gas and air, the "theoretical" mixt. In practical furnace work, more gas is wasted by excess air than by excess gas, although even a 10% excess of gas at  $2500^\circ$  F. means a waste of 18% in fuel. J. L. WILEY

Central generator plant with raw tar recovery. MEYER. *J. Gasbel.* 63, 573-9 (1920).—M. traces the development of the Pintsch water-gas generator culminating with the super-generator type with raw tar recovery perfected during the war for the purpose of supplying fuel oil for the submarines. The design, as shown in a diagram, is a retort superimposed on a generator with revolving grates, thus allowing for low-temp. distn. in the upper part and complete gasification in the lower part with steaming. The gas is drawn off from both sections, that from the retort at a temp. of about  $150^\circ$  containing all of the tar and that from the generator at  $800^\circ$  and tar-free. Lignite briquets, non-coking coal, raw lignite coal, wood and peat can be completely gasified without difficulty. Analyses of the gas from briquets containing 15% water and having a calorific value of 4747, showed for the distn. gas after sepn. of the tar  $CO_2$  11.4%,  $CnHm$  0.4, CO 23.5,  $H_2$  19.2,  $C_2H_4$  1.2,  $CH_4$  5.9,  $N_2$  38.4, calorific value 1967; for the tar-free generator gas  $CO_2$  4.9%,  $CnHm$  0.1, CO 27,  $H_2$  13.2,  $CH_4$  1.4,  $N_2$  53.5, calorific value 1296; giving a mixed gas of  $CO_2$  7.7%,  $CnHm$  0.2, CO 25.5,  $H_2$  15.7,  $C_2H_4$  0.5,  $CH_4$  3.3,  $N_2$  47.1, calorific value 1575. The tar yield was 8.81% with a heating value of 6788 cal. and a C content of about 62.5%. It is also free from naphthalene. The combined gas yield is given as 2.33 cu. m. per kg. of fuel. J. L. WILEY

Manufacture of the higher phenols. "LATEX." *Chem. Trade J.* 67, 439(1920).—Four of the 6 possible xylenols (b.  $215^\circ$ ) are found in coal tar and form part of the "high boiling tar acids." Since the xylenols have a bactericidal value equal to 15 times their vol.  $PhOH$ , and are correspondingly less toxic, they are valuable as antiseptics and disinfectants. The production of this fraction from coal tar is not easy, since the xylenols are very subject to decompn. by heat or in the presence of iron rust or S compds. The mixt. of tar acids sepd. in the usual manner is freed from S compds. by air agitation or treatment with  $PbO$ , and the phenol and cresols distd. off as usual. The residue

from several such charges is distd. in a special still without fractionation and the first runnings added to the cresol fraction. When only half the distillate distils below 208, high-boiling phenols are collected as a greenish mobile liquid, until coking begins in the still. The last runnings, consisting chiefly of creosotes and other "neutral oils," are rejected.

DONALD W. MACARDLE

**Modern by-product oven operation.** S. R. BELLAMY. *Gas Age* 46, 340-2(1920); cf. *C. A.* 14, 2852.—Further operation data and analysis of results from the Ford by-product plant, Detroit. During the last 6 months  $(\text{NH}_4)_2\text{SO}_4$  yield has varied from 25.7 to 28.6 lbs. per ton of coal with volatile in coal ranging between 28.1 and 31%, and speed of operation from 120 to 150 ovens per day. Results show the close relation between sulfate yield and volatile content and contradicts the theory that temps. incident to high-speed operation are unfavorable to  $\text{NH}_3$  recovery. The essential factors for both coke and by-products are: A tight and full oven, proper distribution of heat in flues, back pressure on ovens and main with minimum variation of 1-2 mm., and a definite, daily schedule for pushing. Temp. conditions are directly proportional to the speed of operation and high-speed operation is favorable to high vol. yields of gas. The amt. of gas recovered per ton of coal is from 10-11,000 cu. ft. of which amt. 4500 cu. ft. are used to heat the ovens. The quality of the gas remains uniform at 18 c. p. and 638-660 B. t. u., irrespective of the speed of operation. J. L. WILEY

**Gas coke in relation to cheap power and smoke abatement.** E. W. NICOL. *Gas J.* 152, 209-10(1920); *Gas World* 73, 370-1(1920); *Iron Coal Trades Rev.* 101, 553 (1920).—The introduction of coke in place of coal and of modern coke-burning appliances on the "sandwich system" (*C. A.* 13, 897) has raised the efficiency of many steam plants to a high degree besides eliminating the smoke nuisance, increasing the steaming capacity and output of finished products, and making a substantial financial saving due largely to the higher calorific efficiency obtained. N. concludes that the gas industry holds the key to the problem of obtaining cheap and efficient fuel for power purposes and for the abatement of the smoke nuisance. J. L. WILEY

Petroleum and natural gas (DAY) 22. Biphenylene sulfide in coal tar (KRUBER) 10. Apparatus for mixing small quantities of a gas with other gases in constant proportions (METZGER) 1. Ore dressing and coal washing in 1919 (RICHARDS, LOCKE) 9. Motor alcohol from molasses (CHILB) 16. Ethylene chloride (Brit. pat. 147,908-9) 10.

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JÜPTNER, H. V.: *Beiträge zur Feuerungstechnik*. 1 Teil. Leipzig: Arthur Felix. 189 pp. 10 M.

**Fuel.** JOS. B. MCGRAW. *Can.* 205,334, Nov. 2, 1920. Comminuted carbonaceous material which has been dried to the point of crispness and a binder are mixed and thoroughly masticated until the mass becomes homogeneous. It is then briquetted.

**Gasification of fuels.** F. C. W. TMM. Can. 206,167, Nov. 30, 1920. Fuel is ignited at the top in a closed receptacle, air is drawn downward through the fuel, the gases given off at different periods of the treatment are separately collected and the residue after volatile matter is driven off is used as coke. App. is also specified.

**Gasification of fragmentary fuels.** F. C. W. TMM. Can. 206,168, Nov. 30, 1920. Fragmentary or fine-grained fuels are gasified by passing air downward through the ignited fuel in a receptacle in which the bottom layer of fuel has a smaller cross-sectional area than the body of the fuel and the process is stopped before the remaining fuel is insufficient to reduce the  $\text{CO}_2$  first formed.

**Motor spirit.** C. G. SESTL. Brit. 147,803, July 9, 1920. A motor spirit consists of ethyl, methyl or other alc. or brandy or other alc. liquid mixed with  $\text{CS}_2$  that has been treated with chalk or similar substance.

**Recovering by-products from coal gas.** F. J. COLLIN, AKT.-GES. ZUR VERWERTUNG VON BRENNSTOFFEN UND METALLEN. Brit. 147,633, July 8, 1920. Hot distn. gas is treated in a cooler with cooled ammoniacal liquor condensed during the cooling process, and is washed with a similar liquid in a washer. The liquor and tar collect in a tank in which the tar is sepd., the liquor being forced through a cooler and re-used in the cooler and washer, thus completing the circuit. A portion of the liquor is taken away and supplied to a still where it is treated without the addition of  $\text{CaO}$ . The  $\text{NH}_3$  is either collected as a strong soln. in  $\text{H}_2\text{O}$  or led to a saturator, from which the salt is carried by a siphon to a trickling plane and centrifugal separator. The  $\text{H}_2\text{O}$  from the still containing the fixed  $\text{NH}_3$  is supplied by a pump to a tower where it is cooled by evapn., thence it is pumped by a pump either to the washer and thence back to the still, thus completing a second circuit, or to the cooler to act as cooling agent and back to the cooler, thus completing a third circuit and increasing the percentage of fixed  $\text{NH}_3$  in the  $\text{H}_2\text{O}$ . A portion is from time to time taken away, and, after passing through a regenerative cooler on the still, is conducted to the saturator, the acid in which takes up the  $\text{NH}_3$ ; the  $\text{H}_2\text{O}$  is evapd. and passes out with the  $\text{H}_2\text{S}$  and  $\text{CO}_2$ . It is advantageous to use in the upper part of the washer the fixed ammoniacal soln., and in the lower part the liquor from the cooler also.  $\text{NH}_4\text{Cl}$  can be obtained in marketable form from the  $\text{H}_2\text{O}$  containing fixed  $\text{NH}_3$ , by evapn.

**Gas manufacture.** AKT.-GES. FÜR BRENNSTOFFVERGASUNG. Brit. 148,237, July 9, 1920. Bituminous coal intended for use in Mond gas plant is subjected to a preliminary distn. at a temp. of about  $450^\circ$  in order to remove tarry constituents.

**Gas from bituminous fuels.** Can. 206,165, Nov. 30, 1920. P. KOSTER. Air and steam are alternately forced through incandescent bituminous coal and during the steam supply period sufficient air is blown into the middle of the column of fuel to distil and coke the fresh fuel at the top by burning part of the water gas generated.

**Water gas.** P. KOSTER. Can. 206,164, Nov. 30, 1920. In producing water gas from bituminous coals the air is blown in just above the layer of already coked material, steam is introduced at the lower end of the generator and passes through the entire column of fuel.

**Condensing vapor from natural gas.** J. T. DUFFY. U. S. 1,356,196, Oct. 19. Vapor carried by natural gas is condensed by passing a brush discharge of alternating polarity through the gas, while the temp. is being lowered, thus producing nuclei around which the vapor will condense.

**Ammonium sulfate.** C. SYLL. (FIRM OF). Brit. 147,736, July 8, 1920. Addition to 28,072, 1912. (C. A. 8, 1867.) In the treatment of distn. gas on its way to the saturator, in which the gas is first directly cooled by a counter-current of  $\text{H}_2\text{O}$ , and then directly heated by a counter-current of the same  $\text{H}_2\text{O}$  which circulates between the cooling and heating app., the quantity of  $\text{H}_2\text{O}$  in different temp. zones of the cooler and heater

is so adjusted that the greatest quantity of  $H_2O$  is employed in the zones of highest temp. and the smallest quantity is employed in the zones of lowest temp. An additional intermediate cooling of the gas is employed between the cooler and heater as described in the principal patent, or, alternatively, the circulating  $H_2O$  (or, preferably, that smallest portion of  $H_2O$  which circulates between the zones of lowest temp., is cooled between the cooler and heater. A suitable construction is specified.

**Manufacture of ammonium sulfate from gas liquor.** K. SOTOMURA and MITSUI MINING Co. Japan 35,873, Feb. 23, 1920. In the absorption of  $NH_3$  by dil.  $H_2SO_4$ , the acid is circulated at const. temp. at const. concn.

**Gas generators.** CLARENCE M. CUMMINGS. Can. 205,816, Nov. 23, 1920.

**Gas producers.** FRANK E. MODLIN. Can. 206,136, Nov. 30, 1920.

**Gas producers.** THOS. CLOUSTON. Can. 205,809, Nov. 23, 1920.

**Gas producers.** JNO. F. WELLS. Can. 205,926, Nov. 23, 1920. The producer chamber has an inclined grate beneath a fuel hopper and means for regulating the fuel supply of the grate.

**Gas producers for propelling vehicles.** DAVID J. SMITH. Can. 205,306, Nov. 2, 1920.

**Gas purifiers and regulators.** H. BARNES. Can. 205,385, Nov. 9, 1920.

**Scrubbers, cleaners and coolers for gas.** DAVID J. SMITH. Can. 205,908, Nov. 23, 1920. Structural features.

**Cooling, cleansing and filtering devices for gas.** DAVID J. SMITH. Can. 205,505, Nov. 9, 1920.

**Improvement of furnace using gaseous fuels.** A. C. IONIDES, JR. Japan 35,862, Feb. 21, 1920.

**Apparatus for generation and burning of hydrocarbons.** L. F. BARTOW AND G. CHAMPION KEROSEEN BURNER Co. Japan 35,885, Feb. 27, 1920.

**Resinous material from coal tar.** G. C. BAILEY and F. BOETTNER. U. S. 1,355,103, Oct. 5. A solid or semi-solid bitumen from coal tar is warmed with about 12% of toluene and the mass is centrifuged after cooling until it becomes somewhat stiff. The solids thus obtained are washed with a small amt. of toluene or other solvent and are recovered as a yellow powder which does not darken on exposure to the air, consisting mainly of hydrocarbons m. above  $290^\circ$ . This product may amount to 8% of the original material treated with toluene. The filtrate is distd. to remove the solvent and the residue is distd. under a high vacuum. Up to  $220^\circ$  heavy oil is obtained which does not solidify and may be combined with creosote oils. From  $220^\circ$  to  $275^\circ$  yellowish greases are obtained. From  $275^\circ$  to  $400^\circ$  red resins are obtained, varying in hardness as the temp. increases. These resins may be redistd. The hardest resin may m.  $75-100^\circ$  and is transparent and not discolored on exposure to air and light. The resinous material is sol. in ordinary coal-tar solvents,  $CHCl_3$ ,  $CCl_4$ , linseed oil, cottonseed oil, partially sol. in  $MeOH$ ,  $EtOH$ , glacial  $HOAc$ , petroleum naphthas and ether. The harder distillates flux when carefully heated with S, paracoumarone or paraindene resins, halowax resins, rosin, montan wax and carnauba wax. They may be used in varnish or rubber mixts. or other waterproofing compns.

**Separating constituents of coal tars.** J. M. WEISS. U. S. 1,355,099, Oct. 5. Tar bitumen is treated with  $C_6H_6$ , filtered, the  $C_6H_6$  is distd. off from the soln. and the residue is extd. with petroleum naphtha. The portion of the material insol. in petroleum naphtha may be used for elec. insulating or coating materials where resistance to action of petroleum products is desired, e. g., as tank linings.

**Recovery of tar and ammonia from coal gas.** C. STILL. Can. 206,166, Nov. 30, 1920. The gases are cooled by direct contact with water to sep. the tar; after intermediate cooling the gases are reheated with the water previously used for direct cooling

and then conducted through a satn. bath for absorbing  $\text{NH}_3$ . The gases are then further cooled.

**Recovering constituents of coke-oven and like gases.** C. STILL (FIRM OF). Brit. 147,737, July 8, 1920. The gases freed from tar are subjected to a preliminary treatment when compressed to 20–30 atms. with a suitable absorbent such as alc. for removing in part constituents such as  $\text{CO}$ ,  $\text{H}_2\text{S}$ , and methane. The gases are further compressed to 100–200 atms. in the main compressor;  $\text{NH}_3$  seps. at this point and is recovered as a satd. soln. of  $\text{NH}_4\text{HCO}_3$ ,  $(\text{NH}_4)_2\text{S}$ , etc., by injecting  $\text{H}_2\text{O}$  into the cylinders; the soln. is collected in a tank and the salts sep. on cooling, and the mother liquor may be returned to the compressor or used for washing the gases after they leave each cylinder. The heat of compression is removed in a condenser and the gases then pass to a washer supplied with ammoniacal  $\text{Cu}_2\text{Cl}_2$  soln. for removing  $\text{CO}$  and  $\text{CN}$  as  $\text{NH}_4\text{CN}$ ; from the soln., which collects in a tank, the cyanogen and  $\text{CO}$  are recovered by heating, or the former may be converted into ferrocyanide. Then the gases pass to a washer fed with tar oils, etc., for washing out the benzene hydrocarbons, portions having sepd. as tar oil floating on the liquids in tanks. The  $\text{C}_2\text{H}_2$  and  $\text{C}_2\text{H}_4$  hydrocarbons are absorbed by  $\text{H}_2\text{SO}_4$  in a washer, and the compds. obtained serve for the manuf. of  $\text{EtOH}$  and  $\text{Et}_2\text{O}$ ; the gases first meet in the lower part of the washer  $\text{H}_2\text{SO}_4$  of  $50^\circ \text{Bé.}$  for removing  $\text{C}_2\text{H}_4$ ,  $\text{C}_2\text{H}_2$ , etc., and in the upper part of the washer are afterwards treated with acid of  $66^\circ \text{Bé.}$  for removing  $\text{C}_2\text{H}_2$  and  $\text{C}_2\text{H}_4$ , the acid liquors being collected separately in the compartments; these, like the tanks, are connected to the trunk main by which any escaping gases or vapors are returned to the inlet pipe. The residual gases may be used to supply by expansion energy for driving the compressors; or they may be used for the catalytic synthesis of  $\text{NH}_3$ , in which case any remaining constituents other than  $\text{N}$  and  $\text{H}$  are condensed by cooling the gases to a low temp. in an app. which is conveniently a liquid-air float supplying the necessary quantity of  $\text{N}$ .

**Coking previously so-called "non-coking" coals.** A. ROBERTS. U. S. 1,352,696, Sept. 14. Coals usually classified as "non-coking" such as various Illinois coals in which the amt. of disposable hydrogen does not exceed about 58% of the amt. of disposable oxygen are coked by such rapid heating that the temp. of each portion of the mass is raised to the temp. of decompn. of its resinous constituents to form a binding material before sufficient time has elapsed for their loss by oxidation. In order to permit of such heating, the coal is coked in a retort having 1 sq. ft. of heating surface of the inner wall of the retort itself for each 29–30 lbs. of coal in the retort, heated by flues with 1 sq. ft. of heating area for each 12–13 lbs. of coal in the retort. An extended discussion of the applicability and theory of the process is given.

**Apparatus for producing coke from coal.** F. J. SIVYER. U. S. 1,354,809, Oct. 5.

## 22—PETROLEUM, ASPHALT AND WOOD PRODUCTS

F. M. ROGERS

**Bibliography of petroleum and allied substances, 1917.** E. H. BURKROUGHS. U. S. Bur. Mines, *Bull.* 180, 170 pp. (1920). F. H.

**Petroleum and natural gas.** DAVID T. DAY. *Mineral Ind.* 28, 489–533 (1919).—A review of production in the world's fields, with notes on oil shale and extensive bibliography. A. B.

**The petroleum industry during the war.** KARL GLASER. *Feuerungstechnik* 8, 165–7 (1920).—A discussion of some of the problems met with in Germany during the war. C. J. WEBER

**Petroleum technology problems.** G. A. BURRELL. *Oil Gas J.* 19, No. 21, 84–5 (1920).—Colloid chemists should aid in finding a way to break up oil emulsions. A



good, practical cracking process is yet to be evolved. Economical methods of deeper drilling should be developed to reach deep oil sands. New methods should be evolved of reviving old wells. Coking and cracking of Mexican heavy oil are two processes which need investigation and improvement. The yield of unsatd. hydrocarbons in cracking opens a big field for organic chemists.

R. R. MATTHEWS

Valuation factors of casing-head gas industry. OLIVER U. BRADLEY. *Trans. Am. Inst. Mining Met.* 1920 (advance proof, Sept. meeting), 10 pp. E. H.

Preparation of methanol from lignin. EMIL HEUSER AND HANS SCHMELZ. *Cellulosechemie* 1, No. 6, 49-58(1920).—When 5 g. lignin are heated with 30 cc. 5.7% HCl for 2 hrs. at 5-6 atm. pressure (150-160°) and the residue treated in the same way, 14.83% of MeOH is obtained (practically theor.), besides 2.19% AcOH and 0.73% AcMe. Heating at atm. pressure gave only 2.56% MeOH, while 4 hrs. continuous heating under pressure gave about 13%.

C. J. WEST

Extraction of higher fatty acids and other organic acids from wood tar. E. BOERNSTEIN. *Am. Perfumer* 15, 311(1920).—The crude pine tar, subjected to fractional distn. preferably under reduced pressure, was made to yield palmitic and oleic acids, with a small quantity of "talloleic acid" (liquid abietic acid),  $C_{20}H_{38}O_2$ , described by Fahrion, and certain quantities of the higher boiling (solid) abietic acid known as a constituent of rosin, and finally also arachidic acid. The methods used to isolate the acids in pure form involved their conversion either to esters or Pb salts. The yield of the mentioned acids varies with the origin and nature of the tars, the pine tars of upper Silesia and Poland furnishing 28 g. of oleic and 48 g. of palmitic acid per kg. of tar.

W. O. E.

Turpentine (VEITICH, GROTLISCH) 26. Preparation of alcohol from wood (HEUSER) 16. Oxidation of hydrocarbons with oxygen (KELBER) 10.

Fatty acids from hydrocarbons. H. STRACHE. *Brit.* 147,905, July 9, 1920. The unsatd. aliphatic hydrocarbons contained in mineral oils or the liquid or gaseous products of their distn. or cracking, in natural gas, in the liquid or gaseous products of the destructive distn. of coal, wood, peat, shale, shale oils, or tar oils, or in carburetted water gas, oil gas, water-gas tar, oil-gas tar, lignite tar, producer gas, coal gas, etc., are converted into fatty acids by first treating the gases, oils, etc., with mineral acids ( $H_2SO_4$ , HCl, etc.), whereby the unsatd. hydrocarbons are converted into esters, and then treating these esters with oxidizing agents. For example, the acid sludge or acid resins obtained in refining mineral oils with  $H_2SO_4$  are treated with  $K_2Cr_2O_7$  and  $H_2SO_4$ . Volatile fatty acids formed are distd. off with steam, and the non-volatile fatty acids remaining are freed from  $Cr_2O_3$  by boiling with dil. acid and may be converted into soaps by alkali. Acid anhydrides may be formed in the oxidation process, these also being converted into soaps by alkali. The more volatile acids (butyric, valeric, etc.), may be converted into esters for use in perfumery.

Cracking hydrocarbon oils. STANDARD OIL CO. *Brit.* 147,715, July 8, 1920. High-boiling hydrocarbons, such as fuel oils, are heated to produce hydrocarbons of lower boiling-point such as gasoline by distg. them in a boiler under a pressure of about 4 atm. and passing the vapors, still under pressure, up inclined pipes in which the heavier fractions condense and run back into the boiler. The lighter fractions are condensed in a condenser in which the increased pressure may be retained or not. Cf. C. A. 14, 3788.

Electric cracking of mineral oils. W. JOHNSON. U. S. 1,354,257, Sept. 28. Oil vapors are passed between energized electrodes to effect superheating and cracking of the vapors. An app. is described.

**Apparatus for cracking hydrocarbon oils under pressure.** J. W. COAST, JR. U. S., 1,355,311, Oct. 12. Vapor from a pressure-cracking still is bubbled through a mass of fresh oil and the latter is then fed by gravity to the lower portion of the still through an overflow pipe. Vapors, after thus bubbling through fresh oil being fed to the still, pass through a reflux condenser maintained under pressure and to a  $H_2O$ -cooled condenser.

**Cracking hydrocarbon oils.** J. W. COAST, JR. U. S. 1,355,312, Oct. 12. Oil undergoing cracking is circulated through a highly heated pipe coil under pressure sufficient to prevent vaporization and thence into a large cylindrical still independently heated and maintained under a pressure which will permit vaporization of the oil. Vapors from the cylindrical still are led off to a condenser and liquid oil may be pumped through filters within the cylindrical still and returned to the pipe coil for further heating. Steam or gas or both may be supplied to the pipe coil. A main object of this method is avoidance of injurious coky deposits in the large drum stills.

**Apparatus for cracking hydrocarbon oils.** H. W. JONES. U. S. 1,356,208, Oct. 19. Oil to be cracked is fed from a still or heating app. through a plurality of separately controllable relatively small heated cracking coils from which products pass to a common heater and condenser system.

**Apparatus for dehydration of petroleum.** F. W. MACNIA and P. A. BAULS. Japan 35,858, Feb. 20, 1920.

**Inclined multiple oil-tube still for petroleum oils.** V. I. EMERSON. U. S. 1,356,057, Oct. 19.

**Bituminous compositions.** G. A. HENDERSON. Brit. 148,003, Apr. 30, 1919.  $H_2O$ , petroleum, and S are added to bitumen, or bituminous compns., and the mass is submitted to sufficient heat to dehydrate it and evap. the added petroleum, with the simultaneous injection of Cl. Several modifications are described. Bitumen or pitch is heated to above  $212^\circ F.$ , 10–15% of  $H_2O$  is added, and about 10% by vol. of petroleum premixed with 10% by wt. of the bitumen, etc., of S is added to the mass during ebullition. The mass is agitated and heated under suction to evap. the  $H_2O$  and petroleum, and as much Cl is simultaneously injected as will combine the S (about 33% of the S). In a modification, S is heated with bitumen, petroleum is added and the mass is mixed with a moist vegetable or mineral aggregate. This is heated and agitated, and Cl is injected as before. In a further modification, Cl is passed over heated S to form  $S_2Cl_2$ , which is mixed with petroleum and  $H_2O$ , added to bitumen and the mass is heated to dehydrate it and volatilize the petroleum. Cf. 141,769.

**Decolorizing carbon from woods rich in resins.** M. INADA and the FORMOSAN SUGAR MFG. Co. Japan 35,879. Saw-dust or fine pieces of *Quercus dentata*, etc., from the Arimount in Formosa rich in resins are heated to expel the volatile substances, soaked with 40 parts  $AcOK$  and 80 parts  $H_2O$ , dried, roasted, carbonized at  $450^\circ$  and  $1000^\circ$  for 1 hr. each, crushed, washed with an acid and  $H_2O$ , dried and heated, yielding 20% carbon with large absorbing power.

## 23—CELLULOSE AND PAPER

A. D. LITTLE

**Chemistry of cellulose and its compounds from the colloidal standpoint.** GUSTAVUS J. ESSELEN, JR. *Chem. Met. Eng.* 23, 861–6(1920).—A concise descriptive summary of the structure, elec. and general chem. properties of cellulose, hydration, oxycellulose, mercerization, action of various acids and chem. reagents upon cellulose, viscose, cellulose esters, dope solns., etc. C. J. WEST

**The chemical composition and purification of wood cellulose.** C. G. SCHWALBE *Svensk Pappers-Tidning* 23, 269(1920).—In the attempt to make a cellulose from wood to replace cotton fiber and linen fiber it was found that wood cellulose still held incrustations of substances from the wood itself, together with some decompn. products of cellulose. These crusts contained lignin, sugars, and in cellulose from pine wood resinous substances. No exact detn. for the lignin substances is known, though light is thrown on it by the deepening of color caused by sulfur acids and by a study of the Me number. The sugars may be divided into pentosans and hexosans. The furfural distn. will det. the former. There is no satisfactory method for the latter. The rest of the article seems identical with C. A. 14, 2081. W. SEGERBLOM

**Heat conditions during the digesting of sulfate cellulose.** H. BRAHMER. *Svensk Pappers-Tidning* 23, 269(1920).—The results of extensive investigations are shown in 26 diagrams and graphs. In the Sandberg-Sundblad heat systems part of the heat of the black liquor is used in the form of steam for indirect evapn. in Kestner app. The steam is produced in 2 boilers simultaneously, one stoked with purchased fuel, the other with the dry residue from the black liquor. Part of the steam is led to the evapn. app. from which is obtained low-pressure steam which, mixed with fresh steam, supplies the drying machines. Another part goes to the digesters and the rest is used for incidental purposes. The production of steam in the steam-producing processes associated with the manuf. of sulfate cellulose by the S-S system is essentially a function of the water content of the wood and of the white liquor. The saving of this water, or so-called water economy, is therefore of great importance. Effective water economy can save more than 2 tons of steam per ton of pulp. The digesting process when the escape steam can be saved is insignificantly steam-consuming and may sometimes be steam-producing. The reaction heat produced in the digesting of strong pulp is 1.2% of the heat of burning of the wood detd. by the bomb calorimeter. The specific consumption of fresh steam can be 50% higher in stationary digesters than in rotary digesters. The total consumption of steam in bleached pulp compared with strong pulp is inversely proportional to the yield of pulp. Indirect digesting of strong pulp has no heat-saving quality worth mentioning in the S-S system, if the evapn. app. is sufficiently large and the waste steam can be used. The production of sulfate according to the S-S system can be made self-supporting if the water economy is effectively carried on, and the production can be raised to 60 or 70% in the soda pans. W. SEGERBLOM

**Apparatus for separating cellulose from spinnable bast fiber.** P. WAENTIG. Dresden. *Chem. App.* 7, 145-6(1920), 2 cuts.—The app. consists of a sieve basket of suitable mesh suspended in a tub with overflow adjusted to hold a given depth of  $H_2O$ . The fiber is placed in the basket into which a stream of  $H_2O$  is discharged, the fiber being stirred by a mechanical device while the cellulose passes through the sieve with the  $H_2O$  to a separator where it is recovered. In the large app. the stirring is done with jets of  $H_2O$ . J. H. MOORE

**Investigations relating to digesting wood with alkali.** P. WAHLBERG. *Svensk Pappers-Tidning* 23, 294(1920).—Examn. was confined to colloidal lignin soln. from waste sulfite liquor, to obtain, if possible, some of the sep. constituents. The waste liquor from digesting with alkali was dialyzed in an app. similar to a filter press. Parchment membranes were used and water was run slowly through alternate compartments in a direction opposite to the liquor, thus giving rather concd. solns. Time required was 1 to 2 weeks. The resulting solns. were dark brown, clear and neutral but got muddy on standing. At 50° the soln. becomes gelatinous and a slimy mass containing 95 to 96% water seps. on boiling. The soln. coagulates with small amts. of salts and acids. Electrolysis seps. the bulk at the anode and a small amt. of brown substance at the cathode.  $NaHCO_3$ ,  $NH_3$  and other bases in small amts. dissolve the coagulation

easily and dialysis of this soln. gives the colloidal soln. again. The coagulation caused by heat after washing with warm water and alc. becomes black and friable when dry, also insol. in all usual solvents though alc. acidified with HCl will dissolve it. Further study indicates that the solns. obtained by dialysis do not contain any single substance. The presence of resinous and lignin substances were indicated. The investigation is to be continued.

W. SGERBLOM

The celluloid industry. F. SPROXTON. *J. Soc. Chem. Ind.* 39, 352-3R(1920).

E. H.

Slow-burning celluloid. ANDRÉ DUBOSC AND A. LUTTRINGER. *Bull. soc. ind. Rouen* 48, 91(1920).—From 20% to 35% of cellulose propionate is added to ordinary celluloid. The cellulose propionate has the advantage over the acetate of being sol. in EtOAc.

L. E. GILSON

Cellulose acetates and raw materials. J. R. COCHRANE. *Chem. Eng. Mining Rev. (Australia)* 12, 455-7(1920).—The method of manuf. of cellulose acetate in Australia consists in the action of 33-40%  $\text{Ac}_2\text{O}$  in AcOH upon cotton waste, with 90%  $\text{H}_2\text{SO}_4$  as the catalyst (the max. amt. used is 20% of the wt. of cotton). The fibers lose their strength in 0.5 to 1 hr. and the product can then be mixed by power, the reaction requiring 10-11 hrs. The material is then ripened at constant temp. for 13-15 hrs. until the sirup is homogeneous, and then hydrated by adding 50% AcOH to the sirup, between 80-120 g. per 100 g. cellulose being used. This occupies 12-16 hrs. The required acetate is plastic in  $\text{CHCl}_3$  and sol. in AcMe, and contains about 53% AcOH. Pptn. is effected by addition of  $\text{H}_2\text{O}$ .

C. J. WEST

The aldehyde content in sulfite liquor. R. SIEBER. *Svensk Pappers-Tidning* 23, 336(1920).—Following Neuberg's suggestion that sugar when fermenting may form acetaldehyde and glycerol, expts. were done to det. the alc. content and the aldehyde content of fermented sulfite liquor of different  $\text{SO}_2$  contents. Long tables of data show that liquors weak in  $\text{SO}_2$ , whether org. or inorg. compds., yield less aldehyde than those rich in  $\text{SO}_2$ . Liquors from digesters of bleached pulp are more favorable than those from strong pulp digesters. The amt. of aldehyde in the wort depends on the quality of the pulp and the methods of digesting. Pulp from different districts give different amts. of alc.

W. SGERBLOM

Control of analysis of the acid from digesting sulfite. P. KLASON. *Svensk Pappers-Tidning* 22, 227, 255(1919).—The acid from digesting sulfite consists of a soln. of neutral  $\text{CaSO}_3$  in S acids and must therefore be considered as containing  $\text{Ca}(\text{HSO}_3)_2$  and the excess  $\text{SO}_2$ . That part of the acidity belonging to the  $\text{CaSO}_3$  is called bound  $\text{SO}_2$ , the rest free  $\text{SO}_2$ . Lengthy tables give the results of analyses designed to check up the accuracy of different methods in use, particularly where those methods seem to give results not in agreement. These show that both Winkler's and Koehn's titration methods rest on correct assumptions, that the alk. reaction of  $\text{Na}_2\text{SO}_3$  does not apparently affect the titration, and that both methods therefore give identical results. The errors therefore must be ascribed to the analyst, his titration solns., his measuring app., his readings, and not least his taking of samples. Samples for analysis must never be taken by suction but by pressing the acid up into the pipet. The digesting liquor after standing a while does not have the same compn. at the bottom as at the top, there being less free  $\text{SO}_2$  at the bottom than near the surface. The taking of samples is of the utmost importance and cannot be done outside the factory.

W. SGERBLOM

Some observations on waste sulfite liquor. P. WAHLBERG. *Svensk Pappers-Tidning* 22, 257(1919).—Solid substances are not pptd. from waste sulfite liquor of 35° Bé. by fine NaCl though pptn. occurs in less concd. solns. Four g. of NaCl is the smallest amt. which will ppt. 1 g. of NaCl-free sediment from a liquor holding about

40% solid substances. The pptd. org. substances diminish with the increase in the  $\text{SO}_2$  content in the liquor. Substances containing S can be obtained by dialysis using colloidion as the film. A combination of spectroscopic analysis and titration analysis has been devised for the colored soln. obtained by dialysis. W. SEGERBLOM

The reason for the incrustations in the heaters and evaporators with means to prevent them, particularly with reference to the evaporation of sulfite liquor. R. W. STREHLENERT. *Svensk Pappers-Tidning* 23, 190(1920).—Sulfite liquor is one of the hardest liquors to evap. because of the Ca salt deposited. Attempts to prevent the deposit of pptd. salt by rapid circulation of the liquor failed. The production of insol. substances in the scale seems due to the chem. reaction taking place in the liquor during the heating. Prevention of such reactions may prevent formation of scale. The incrustation from sulfite liquor consists mainly of  $\text{CaSO}_4$  and  $\text{CaS}$ ; smaller amts. of  $\text{CaSO}_3$ ,  $\text{SiO}_2$  and org. substances are present. The first-named is probably due to the oxidation of the S acid. It is therefore better to use neutralized liquor.  $\text{NaHCO}_3$  has been used successfully, for the resulting salts are sol. If the liquor containers are kept full or the walls above the liquor are kept moist there is less scale. Passing an electric current through the container was studied with promising results.

W. SEGERBLOM

Composition of the sulfite coal (Strehlenert). HANS KRULL. *Cellulosechemie* 1, No. 6, 58-59(1920).—Two samples showed the following analyses: Moisture, 9.24, 6.34; ash, 9.08, 35.45; C, 54.64, 34.66; H, 4.77, 4.10; S, 9.04, 15.25; heating value, 5424, 4151; heating value (ash-free and dry, 6700, 6000). The first analysis gives a formula  $(\text{C}_{16}\text{H}_{11}\text{SO}_3)_x$ , the second  $(\text{C}_{11}\text{H}_8\text{S}_2\text{O})_x$ . When this is compared with the formula for the sulfonic acid of lignin,  $\text{C}_{40}\text{H}_{40}\text{O}_{17}\text{S}_2$ , it is evident that a considerable carbonization of the sulfite liquor has occurred. C. J. WEST

Non-rusting metals for paper manufacturers. ANON. *Svensk Pappers-Tidning* 23, 192(1920).—Probably no industry that uses Fe and steel is so troubled with rusting and erosion as the paper industry. Fe rust and Fe salts cause the greatest damage in paper making. The substitution of  $\text{Al}_2(\text{SO}_4)_3$  for mineral alum has increased the trouble. The different kinds of acid-resisting Fe and non-rusting steel are compared and paper manufacturers are urged to substitute these for the more expensive non-ferrous metals now used in paper machinery. W. SEGERBLOM

Nature and use of lubricants (WENIGER) 13. Preparation of methanol from lignin (HEUSER, SCHMELZ) 22. Solvent for pyroxylin (U. S. pat. 1,356,440) 26.

Cellulose fiber. J. A. DECEW. U. S. 1,354,731, Oct. 5. Lignocellulose is impregnated under pulp-cooking temp. and pressure within a digester with hot 15% caustic alkali soln. and the excess of the soln. is withdrawn after the material is satd. so that the lignone will be attacked only by the alkali absorbed. This serves to secure uniform action without circulating the soln.

Cellulose solutions. Z. OSTENBERG. U. S. 1,355,415, Oct. 12. Cellulose solns. suitable for prepg. cellulose esters or making films are prepd. by mixing the cellulose, e. g., wood or cotton, with a solvent formed by mixing  $\text{CaCl}_2$  and  $\text{H}_2\text{SO}_4$  of over 60% concn.

Cellulose nitrate composition. S. J. CARROLL. U. S. 1,354,725, Oct. 5. Tetrachloronaphthalene and triphenyl phosphate are colloidized together and the resulting mixt. is then combined with a soln. containing cellulose nitrate and fusel oil to form a material for films or varnish. U. S. 1,354,726 relates to a similar compn. in which cellulose acetate is used, together with the cellulose nitrate and other ingredients.

Nitrocellulose film. J. G. JARVIS. U. S. 1,354,401, Sept. 28. Sheets of low

combustibility are formed of nitrocellulose, acetylcellulose, an acetone collodion, chloral hydrate and cinchonine.

**Viscose.** L. LILIENFELD. U. S. 1,355,985, Oct. 19. Cellulose not previously hydrolyzed is treated with a soln. containing an amt. of caustic alkali corresponding to materially less than 36 parts by wt. NaOH per 100 parts of cellulose and viscose is produced by the action of  $\text{CS}_2$  on the alkali cellulose thus formed.

**Molding cellulose acetate.** DR. A. CELLON-WIRKER. Brit. 147,904, July 9, 1920. Cellulose acetate is subjected in a finely divided state, preferably in the presence of very small amts. of low-boiling or volatile substances, to a high pressure at a temp. approaching the decompn. point and allowed to cool under pressure. The operation may be carried out at a lower temp. and pressure by adding 2-5% of  $\text{H}_2\text{O}$ , alc., etc., or solid substances such as naphthalene or camphor. Preferably the material obtained by a first pressure is disintegrated and subjected to a second pressing operation. In this case, the cooling under pressure may be omitted by providing the mold with channels, nozzles, or slots of small diam., through which the hot material is forced under pressure in the form of filaments or ribbons. The disintegrated material may be mixed with large quantities of mineral powders, e. g., 2-5 times the amt. of cellulose acetate; before applying the final pressure. The material may be molded with inlays of wood, ebony, etc.

**Readily nitrated wood fiber.** C. CLAESSEN. Ger. 300,069, July 29, 1915. Uniform, highly porous and readily nitrated cellulose may be obtained at a low cost by drying the pulp as it runs from the machine and rolling it or pressing it to a compact mass which is then comminuted to form threads or strips. The product can be nitrated either in centrifugal machines or in nitrating pots.

**Coated vulcanized fiber.** M. AROSIO. U. S. 1,355,586, Oct. 12. A film of celluloid-like acetylcellulose compn. is applied to vulcanized fiber in order to waterproof it.

**Water-resisting coating adapted for use on paper or textile fabrics.** G. W. MILES. U. S. 1,354,575, Oct. 5. A coating suitable for use on paper or cloth is prepd. by saponifying air-oxidized rosin or other oxidized resin with ammonia and dilg. the soln. with  $\text{H}_2\text{O}$  at ordinary temp.

## 24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROE

**Production of explosives in the United States during the calendar year 1919.** WILLIAM W. ADAMS. Bur. Mines, *Tech. Paper* 259 (1920).—This paper covers the classification of explosives; production and distribution for each year from 1912 to 1919 inclusive; fatalities in the mining industry from explosives; exports for each year from 1913 to 1919 inclusive; and list of permissible explosives tested to May 31, 1920. The production for 1919, including exports, was black blasting powder 180,511,050 lbs., permissibles 38,854,832 and "high" explosives other than permissibles 198,268,588 or a total of 417,634,470 lbs., which was 81,490,190 lbs., or 16% less than in 1918. With the increased use of permissible explosives the fatalities from explosives in bituminous coal mines have diminished from 9.8% in 1903 to 3.4% in 1919.

CHARLES E. MUNROE

**Smokeless powder.** A. LUTTRINGER. *Bull. ind. soc. Rouen* 48, 89-90 (1920).—Nitrocellulose is gelatinized by the addition of 4-15% of Me or Et ricinoleate.

L. E. GILSON

**The type of explosive in the Wall Street explosion.** JOHN R. MARDICK. *Chem. Met. Eng.* 23, 717-8 (1920).—Smokeless powder burns and explodes without smoke or

residue, giving a light yellowish flame. TNT and picric acid burn and explode with a deep orange-yellow flame and dark fumes, but no residue. When exploded in the open loose particles of TNT and picric acid are carried into the air and color the fumes yellow. In the Wall St. explosion a residue was scattered over the ground and on the window weights that had come in contact with the explosive matter. Eliminating the above explosives leaves black powder, which is a weak explosive and leaves behind such a large cloud of black smoke it can be easily recognized; dynamite and nitro starch explosives, which leave residues consisting of  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{SO}_4$ , and other S and Na compds. if S was used in the manufacturing process. Chlorate powders leave Na and K carbonates, sulfates, chlorides and sulfides. The evidence shows that either the explosive was not powerful or the quantity used was very small; not exceeding 50 lbs. and probably nearer 25 lbs. The latter weight of dynamite would do more damage than was done. It would make a big cavity in the street just under the wagon in spite of the explosive being placed 3 ft. from the ground. An important point on which all witnesses agreed was as to the length of the flame which burned shades at windows on a seventh story. M. holds that a chlorate explosive is the only one having such a long flame, and he concludes that the basic ingredient used in the explosive was a chlorate; that the explosive was not well made but concocted by the plotters of the crime from such ingredients as might be purchased in small quantities from chemical houses, drug stores, etc.; and that it was not confined in any stout metal container or bomb but was probably put in a wooden box and covered by iron slugs and window weights.

CHARLES E. MUNROE

The behavior of time fuses. A. V. HILL. *Nature* 106, 214-5(1920).—The "powder train" time fuse consists essentially of one or more rings of highly compressed gunpowder forced into a metal groove. The ring is ignited by a cap at the moment the shell starts from its seat and after the detd. portion of the ring has been burned it ignites a pellet that explodes a detonator which detonates the charge in the shell. The "fuse setting" which dets. the length of the powder ring to be burned and hence the time the fuse is to be burned, is effected by turning a metal ring around an axis parallel to that of the shell. The combustion gases escape from a hole in the fuse usually at the side but sometimes in the nose of the shell. In the past "powder train" fuse was used principally in shrapnel, where the target was usually large and at rest, or so nearly so that "ranging" could be employed, and it gave remarkably good and consistent results, but with the introduction of anti-aircraft guns for use against small and swiftly moving targets defects were developed due to the effects of variations of air pressure, velocity, spin and temp. In a fuse at rest the time of burning is proportional to the length of the powder train burned and the rate of burning is a function of the atm. pressure upon it. In a fuse fired in a shell the rate of burning is usually greatest at first when the velocity of the shell is highest and decreases gradually as the velocity of the shell decreases until a nearly const. value is attained. But in some instances fuses from the same lot may act in the above manner when used in one type of shell and in the reverse manner when used in another type of shell. The relation of time of burning and length of powder train burned is explained by the theory of "dynamic pressure" at the escape holes which holds that as the shell moves through the air the pressure at any given point varies with the speed and at any given speed varies from point to point on the shell. So completely has this theory been verified that an observed relation between "fuse setting" and time of burning has been satisfactorily used to det. the pressure at various points on the head of a shell moving at various speeds up to 1600 ft. per sec. The "dynamic pressure" may have a negative sign, as when the escape hole is too far back from the nose of the shell or under the lee of a projection on the fuse, and if sufficiently large the powder may refuse to burn and the shell will be "blind."

In trials of fuse of the same lot fired in shell to various heights up to 20,000 ft. from 5 different 3-in. guns which differed only in their muzzle velocities most erratic results were obtained, to explain which the theory of "spin" was advanced and verified by tests on shell "at rest," i. e., without forward translation, up to 30,000 r. p. m. and it was found possible to double the rate of burning of a fuse or to prevent its burning by the spinning. The explanation is found in the centrifugal effect on the slag produced by gunpowder in its combustion. When the spin is high the gunpowder, warmed, softened and just ignited by the combustion of the previous layer, is "spun" outwards to the outer edge of the groove before it has had time to burn properly and to ignite the next layer. Consequently combustion is slowed down and may fail altogether. The absence of any effect from spin in the case of a special powder giving no slag, as well as the fact that "blind" fuses are found to have failed first on the *inside* edge of the ring confirm the opinion that the centrifugal effect on the slag is the prime cause of the trouble. The centrifugal effect varies as the square of the spin and when rotations of 30,000 r. p. m. are reached it is almost impossible to attain any accuracy. An obvious method of reducing the spin is by reducing the number of turns in the rifling of the gun. Another factor affecting the behavior of fuse, though a minor one, is temp. This may be less with the shell in motion than "at rest." Hence in testing fuses "at rest" the fuse is rotated in a box at any desired "spin" and pressure while the box is subjected to a spray of water to maintain a constant temp. (Comment. Many failures of ammunition have been attributed to carelessness in manuf. of the explosive or to substitutions of other material than that called for. Manufacturers of explosives may note that such failure may have been due to defects in the device used or to its use under disadvantageous conditions.)

CHARLES E. MUNROE

American production of military high explosives and their raw materials. CHARLETON H. WRIGHT. *U. S. Nav. Inst. Proc.* 46, 1561-99, Oct. 1920.—Graphs are given for the production in the U. S. of TNT, picric acid,  $\text{NH}_4$  picrate, tetryl,  $\text{NH}_4\text{NO}_3$ , Trojan powder,  $\text{Hg}(\text{ONC})_2$ ,  $\text{HNO}_3$ ,  $\text{C}_6\text{H}_6$ ,  $\text{C}_2\text{H}_6$ ,  $\text{C}_6\text{H}_5\text{OH}$  and  $\text{NH}_3$  for 1918,  $\text{H}_2\text{SO}_4$  for 1904-1918, beehive and by-product coke for 1892-1919 and total production of military high explosives for 1918. For most of the explosives the quantity of each kind of raw material required in its manuf. of unit quantity is stated and the maintenance of dyestuff and other chem. plants for protection in war is urged.

CHARLES E. MUNROE

Calculations involved in the preparation of acid mixtures used in the manufacture of nitrocellulose. WILLIAM YOUNG. *J. Soc. Chem. Ind.* 39, 315-67 (1920).—An algebraic method used is demonstrated by examples. In use in a large explosives factory it has been found less laborious than the arithmetical method and more adaptable to changing conditions than the graphical method. With the use of certain justifiable assumptions it may be used in the construction of tables from which the proportional parts may be taken.

CHARLES E. MUNROE

Ammonium perchlorate. UGO ALVISE. *Rass. min.* 57, 21-2, 43-5, 73-4, 88-91 (1920).—An array of evidence, chiefly from patent literature, in defense of A.'s claim to priority in the use of  $\text{NH}_4\text{ClO}_4$  as an ingredient of explosives. Confusion has arisen in several cases through the failure of writers to distinguish  $\text{NH}_4\text{ClO}_4$  from  $\text{KClO}_4$  and  $\text{KClO}_3$ .

M. R. SCHMIDT

Investigation into the causes of the self-ignition of ether-air mixtures. J. A. McCLELLAND AND H. V. GILL. Dublin. *Sci. Proc. Roy. Dublin Soc.* 16 (N. S.) 109-19 (1920).—When an ether-air mixt. is allowed to expand from a reservoir into an exhausted tube, a flame is sometimes observed to start in the tube and travel backwards toward the reservoir. It was found that the proportions of  $\text{Et}_2\text{O}$  and air were not important, so long as there was sufficient of the former; that the presence of dust



had no significance, and that the humidity of the mixt. was not important; the pressure of gas in the reservoir could also be varied within wide limits without any great effect. However, when the pressure in the exhausted tube exceeded 5 cm. of Hg, no ignition took place. The explosion tube was 2-3 inches in diameter and 3-8 feet long; no ignition occurred in tubes less than 2 ft. long. Various obstacles in the closed end of the tube, such as cotton wool, also prevented ignition. Since it seemed that the observed effect was due to temp. rise, the tube was explored with a thermocouple while air alone was admitted from the reservoir. The temp. rise was greatest for tubes about 8 ft. long, and showed a sharp max. about 5 cm. from the closed end. It was also a max. for about 100 cm. difference in pressure between the air in the reservoir and that in the tube. The actual max. temp. reached by the air was 185-193°, and since such a temp. is sufficient to ignite the ether-air mixt. the cause of the explosions is clear. The rise in temp. to a max. near the closed end is probably due to the conversion of the translational energy of the gas stream into heat, especially where it meets the portion reflected back from the end of the tube.

W. B. V.

**Explosions from fish meal dust.** DAVID J. PRICE. *The American Fertilizer*, Nov. 20, 1920.—Though no accidents from fire or explosion have been reported during manuf., yet investigators of the Bureau of Chemistry, Dept. of Agriculture, found the operation of grinding dried fish to be so dusty a one that tests of this dust seemed advisable. These showed that fish meal dust is very inflammable and forms mixts. with air which explode with much violence.

CHARLES E. MUNROE

**A disastrous explosion of aluminium dust.** DAVID J. PRICE. *Chem. Met. Eng.* 23, 915-19(1920).—The explosion occurred Feb. 26, 1920, in the "satin-finishing" dept. of an establishment engaged in mfg. cooking utensils. In this operation much Al dust was produced and hence an exhaust system had been installed for removing dust from the room, in which some 55 persons were employed. The explosion was attributed to a piece of No. 7 B & S Fe wire about 8 ft. long which had been introduced from the outside through the discharge end of the exhaust pipe, wound about the blades of the exhaust fan and struck sparks by friction against the walls of the exhaust pipe as the wire revolved with the fan. Cf. C. A. 14, 2988.

CHARLES E. MUNROE

**Inflammability of jets of hydrogen and inert gas.** P. G. LEDIG. *J. Ind. Eng. Chem.* 12, 1098-1100(1920).—The purpose of the investigation was to det. the max. amt. of H which could be used with He in balloons without losing the advantage of non-inflammability. The tests were made by the blow-off method through orifices of 5-30 mm. diam. in plates 2 mm. thick. It was concluded that, under the most favorable conditions, a jet of He containing more than 14% of H can be ignited in the air. 18-20% of H may be mixed with He, without producing a mixt. which will burn with a persistent flame when issuing from an orifice under the conditions prevailing in balloon practice. More than 20% of H in a H-He mixt. cannot be used in a balloon without sacrificing safety from fire.

CHARLES E. MUNROE

**Improperly handled gas cylinder explodes with fatal results.** B. W. DUNN. *B. E. Accident Bull.* 49, Oct. 1, 1920.—A supposedly empty cylinder which really contained a quantity of compressed CO<sub>2</sub> was stored in a freight house from July 1 to 7 in an upright position. When moved on the latter date it was dropped on the cement floor when it exploded violently, the cylinder being ruptured and a fragment of it killing instantly the man handling the cylinder. It is claimed this explosion would have been prevented if the cylinder had been stored on its side as required in I. C. C. Rule 1893 (d).

CHARLES E. MUNROE

**Fire while breaking down thermalloy.** T. H. CROZIER. Special rept. 237 H. M. Insp. Exp. 1920.—Thermalloy is a mixt. of Fe<sub>2</sub>O<sub>3</sub>, 50, Al 27, and S 23 parts which was largely used in the war in incendiary bombs, it being made active by an igniting mixt.

composed of Mg, Al and S. The surplus material left by the cessation of active military operations was being salvaged by passing it, in granular form, through a magnetic separator which allowed the Al and S to fall in one receptacle and carried the  $\text{Fe}_3\text{O}_4$  to another. The accident occurred on Aug. 14, 1920, at Kingsway Works, Linton, Bedfordshire, when there was present on the premises 250 tons of thermalloy, 20 tons of smoke mixt., and 23-24 million rounds of small-arm ammunition, and is attributed to fire due to some electrical cause, probably connected with the magnetic separators and not necessarily due to an electrical breakdown, since it might have been caused by thermalloy dust finding its way inside the revolving brass covering of the magnets. A few secs. after the first warning the whole bulk of the thermalloy took fire and burned with almost explosive violence, flames rising to a very great height, the sound being variously described as a dull booming like a far-off gun and as a rushing roar, but there was no real explosion as proved by the absence of a crater and that a light building 40-45 yard distant was not wrecked. But the rush of gas was enormous and sufficient to hurl heavy masses of brickwork to a distance of 30 yds., while the heat was such that heavy steel girders were twisted, bent and melted, in places. The fields all about for a distance of some hundreds of yards were covered with small metallic-looking globules, probably of  $\text{FeS}$ .

CHARLES E. MUNROE

Inflammability and explosibility of benzene and benzene (OSTWALD) 21.

Progressively burning explosive powder. R. G. WOODBRIDGE, JR., K. K. V. CASEY and C. I. B. HENNING. U. S. 1,354,442, Sept. 28. An explosive suitable for use in rifles is formed of a deterrent-carrying nitrocellulose powder in granular form carrying a firmly adhering coating of an easily ignitable propellant such as black powder and dinitrotoluene.

Potassium chlorate. E. R. WOLCOTT. U. S. 1,355,203, Oct. 12.  $\text{KClO}_4$ , in the form of silky or fibrous crystals is obtained by crystn. from an aq. soln. of  $\text{KClO}_3$  and sulfonated derivs. of Cal. petroleum. The product is adapted for use in explosives and is less likely to explode during grinding with combustibles than the ordinary  $\text{KClO}_3$ .

Nitrocellulose explosive powder coated with black power. R. G. WOODBRIDGE, JR. U. S. 1,354,640, Oct. 5. A propellant powder is formed of nitrocellulose powder grains coated with black powder which serves to facilitate ignition.

Nitrocellulose explosives. CHEM. FABRIK VON HEYDEN, AKT.-GES. Ger. 302,460, July 17, 1917. Heretofore, in the manuf. of explosives from nitrocellulose, pure acetone has been used. As a substitute for acetone mixts. of furfurole with  $\text{CH}_3\text{OH}$  have proved satisfactory, and for certain nitrocelluloses, mixts. of furfurole with formic and acetic esters, ether, benzene or  $\text{CHCl}_3$  have been used.

Mixing solids with liquids; nitrating cotton. V. SMEW. Brit. 148,124, July 9, 1920. App. for dissolving materials in or impregnating them with acids, etc., particularly applicable to the nitration of cotton, comprises a receptacle for the materials—e. g., cotton and acid, a reciprocating and rotating plate which depresses the cotton into the acid and which, during its immersion, agitates the materials, and a scraper which engages the plate after it leaves the receptacle; the reciprocating motion of the plate is controlled automatically. A suitable construction is specified.

Nitrating cotton, etc. V. SMEW. Brit. 148,125, July 9, 1920. App. for nitrating cotton, etc., comprises a series of nitrating vats carried on a circular table which is rotated to bring the vats in succession first beneath measuring devices from which they receive their charge of acid and material to be nitrated, and then beneath immersion and agitating devices, e. g., as described in 148,124 (above) for depressing the material into and agitating it with the liquid, and finally at the end of their travel to bring

the vats opposite to centrifugals into which their contents are tipped; the table is stopped automatically at each step of its movement, and the starting is effected by a hand lever. A suitable construction is specified.

## 25—DYES AND TEXTILE CHEMISTRY

L. A. OLNEY

**Monoazo dyestuffs suitable for the production of colored lakes and pigments.** W. ALLSEBROOK. *J. Oil Color Chem. Assoc.* 2, 126-40(1919).—A review. A systematic scheme for the qual. analysis of mono-azo dyes can be built up on their characteristic color reactions with concd.  $H_2SO_4$ , and with reducing agents. The latter splits these dyes at the union between the 2 N groups into the original amine, and an amino deriv. of the compd. with which the diazo group was coupled. This reaction can be used for the volumetric estn. by titration with  $TiCl_3$ , the coloring matter acting as its own indicator, and each azo group requiring  $4TiCl_3$  for complete reduction. A. gives a brief description of the aminoazo and hydroxyazo dyes, their sulfonic acid derivs., and uses in making lakes; and reviews patents and urges English manuf. of intermediates for making lithol red R, lithol scarlet, Helio fast red, etc. Discussion.

F. A. WERTZ

**Table for the identification of dyes by means of the spectroscope.** JAMES MOIR. *J. S. African Assoc. Anal. Chem.* 3, 9-13(1920).—This table consists of 3 columns, the first being a number for the dye as given in A. G. Green's edition of Schults and Julius, 1904 or 1908, the second the name and solvent of the dye, and the third the wave length numbers ranging from 668 to below 400. The number of wave length readings recorded was 107. The technic of making an observation consists in dilg. the dye soln. until its absorption band is narrow as possible, and not opaque; sometimes a diln. of 1 to 2 thousand is necessary with powerful dyes. The slit should be narrowed to focus the instrument, then widened until the Fraunhofer lines are quite indistinct, and the index set to agree with the center of the hazy image of a Fraunhofer line. The instrument should point to clear sky, plenty of light being essential. Dyes which change color with acid or alkali were observed and recorded in both conditions; thus trypan blue gives 583 when neutral and 596 when alk.

L. W. RIGGS

Alkaline melts for chemical reactions (U. S. pat. 1,354,451) 4. Water-resisting coating for paper or textile fabrics (U. S. pat. 1,354,575) 23.

GEORGIEVICS, GEORG VON AND GRANDMOUGIN, EUGÈNE: **A Text-book of Dye Chemistry.** Translated and revized from 4th German Ed. London: Scott, Greenwood & Son. 560 pp. 30s. net.

**Dyes.** L. CASSELLA & Co. Brit. 147,703, July 8, 1920. Vat dyes are obtained by heating with alkali sulfides the condensation products from *m*- or *p*-nitroaniline, or their substitution products, and benzoquinone or its halogen derivs.; preferably  $(NH_4)_2S$  is employed in aq. or alc. soln. and the reaction is effected under pressure. The products dye wool from a vat various shades of gray to black.

**Dyes.** L. CASSELLA & Co. Brit. 147,704, July 8, 1920. Vat dyes are obtained by condensing arylamines with the thio derivs. of benzoquinone, or its halogen derivs., described in German Specifications 175,070; suitable thio compds. are quinone mono- and dithiosulfonic acids, quinone mercaptans, diquinone mono- and disulfides, and their halogen derivs. The condensation products obtained when the reaction is carried out in an inert diluent or solvent, such as  $H_2O$ , alc. or acetone, dye wool yellow to yel-

lowish brown shades from a hyposulfite vat; products dyeing fuller shades are obtained by carrying out the condensation at raised temps. in an acid medium, such as glacial or dil. HOAc, or formic acid, or by treating with acids or acid reducing agents the condensation products obtained in an inert medium. The condensation may be facilitated by adding an O carrier, such as a cerium salt, and blowing in air.

**Dye of the magenta type.** J. L. KANE. U. S. 1,355,048, Oct. 5. In producing magenta, aniline 14 is mixed with toluidine 54, dried pulverized starch paste 20 and concd. aq. HCl 67 parts, the mixt. is heated to about 130° and there is then added to it aniline 8, toluidine 26 and PhNO<sub>2</sub> 55 parts, and when this mixt. is at a temp. of about 100° a soln. formed of Fe 3 parts dissolved in HCl is slowly added. The mixt. is heated under a reflux condenser for 6-8 hrs. at 180°, aniline and toluidine are driven off by steam distn., the resulting product is poured into 500 parts of boiling H<sub>2</sub>O, acidified with HCl, treated with NaCl and boiled. The soln. is poured off, the residue is allowed to cool, forming a green brittle mass which is extd. with a large amt. of boiling H<sub>2</sub>O acidulated with HCl and this soln. is allowed to cool to 60° and salt is added to it to sep. the dye. The magenta thus formed is stated to have an unusually good depth and fastness of color.

**Cymene dyes.** C. E. ANDREWS. Can. 204,769, Oct. 12, 1920. Aminocymene is dissolved in water containing HCl at 10° or lower and a soln. of NaNO<sub>2</sub> is added while stirring and maintaining the low temp. to produce 1-methyl-4-isopropyl-2-diazonium chloride. This soln. is added to β-naphthol dissolved in dil. NaOH at 10° while stirring, thus producing the Na salt of aminocymeneazonaphthol which is a dye of a very brilliant reddish orange color. Any suitable hydroxy aromatic compd. may be used instead of β-naphthol. Cf. C. A. 13, 2765.

**Dimethyldiisopropylbenzidine azo dyes.** C. E. ANDREWS. Can. 204,774, Oct. 12, 1920. A dye comprizing the tetrazo compd. of dimethyldiisopropylbenzidine coupled with a hydroxy aromatic compd. is prepd. by dissolving the dimethyldiisopropylbenzidine in hot water containing HCl and NaOH and cooling the mixt. to about 10°. A further quantity of HCl and a soln. of NaNO<sub>2</sub> is added to produce the tetrazo deriv. This deriv. is added to water containing salicylic acid and NaOH while stirring and maintaining the temp. at 10°. The reaction requires about one day, during which a further quantity of NaOH is slowly added. The liquid is allowed to settle and the dye seps. in the form of a yellowish brown powder. Cf. C. A. 13, 2766.

**Dyeing fabrics.** P. KAUFMANN. Brit. 148,149, July 9, 1920. A machine for dyeing linen, cotton, and union goods with vat and S dyes consists of a vat or vats into which the goods are passed, either directly or from a feeding tank, so that when thoroughly wet and free from air they sink to the bottom of the vat. This movement is facilitated by the method of heating the vat, which consists in placing a steam pipe behind a sliding panel in the vat. The upper and lower parts of the panel are perforated to allow the liquids to circulate through the vat. The feeding tank containing the dye is used in dyeing with light shades, and with indigo; thence the fabric passes through squeezing rollers and to the fixing vat, sinking to the bottom, which is curved. A suitable construction is specified.

**Dyeing raw fibrous stock.** P. MACINTYRE. U. S. 1,355,797, Oct. 12. In dyeing cotton or similar raw stock, the material is subjected in bulk to the action of a dye liquor such as indigo and the dye is then oxidized by forcing air under pressure through the mass.

**Producing marbled patterns on textiles, etc.** C. JAGER GES. and R. W. CARL. Brit. 148,187, July 9, 1920. A film of aniline dye is floated on the surface of a liquid, preferably by sprinkling with a brush, and arranged in the desired patterns by means of a splinter of wood, a comb, etc.; the material to be treated is then applied to the

surface of the liquid and the dye adheres to it. The most suitable dyes are those which are sol. in fats, *e. g.*, the "cerotin colors" or mixts. of these with color bases. For treating textiles, solvent oil is the most suitable solvent and for treating sheet metal, zapon varnish. Examples are given of the treatment of a textile, paper and sheet metal with different dyes.

Apparatus for dyeing yarn or thread. J. A. FIRSCHING. U. S. 1,356,059, Oct. 19.

Apparatus for scouring or dyeing wool. H. WALKER. U. S. 1,354,081, Sept. 28.

## 26—PAINTS, VARNISHES AND RESINS

A. H. SABIN

Turpentine: its sources, properties, uses, transportation and marketing, with recommended specifications. F. P. VEITCH AND V. E. GROTLISCH. Bur. Chem., U. S. Dept. Agr., *Bull.* 898, 1-51(1920). W. H. ROSS

Some new physical apparatus. A. E. BAWTREE. *J. Oil Color Chemists Assoc.* 3, 109-30(1920).—An app. particularly adaptable for *detg. sp. gr. of heavy paste paints*, overcomes usual inaccuracies in reading the vol. of displaced  $H_2O$ , by having a narrow graduated tube closed at one end and attached at the other to a larger brass cylinder provided with a lid. After taking a blank reading on the graduated tube, the instrument is inverted, a shallow basin containing a weighed amt. of paste is attached inside the lid of the cylinder, the app. is reinverted and the rise in level of the  $H_2O$  read. Calcn. of sp. gr. from the sp. gr. of the pigments and liquids used in the pastes, is open to error because of the possibility of vol. changes on mixing. Detn. of viscosities of paint products by any of the usual viscometers is of no real value. Tabulated objections to the Redwood and Doolittle instruments appear to be overcome by a newly developed Hadfield-Bawtree *viscosimeter*, which consists of a Cu vessel of about 150 cc. capacity provided with an airtight cover through which a tube 1 in. in diam. and of a capacity of  $(50 \times 3.1417 \div 4)$  cc. The lower end of this tube reaches nearly to the bottom of the Cu vessel and has a 1-mm. orifice with a ball valve; the upper end of the tube is open to the air. The cover is also provided with a 2-way stopcock. For a detn. the outer vessel is nearly filled with the liquid, the cover tightened, definite air pressure applied, and the time noted for filling the tube through the orifice. In this instrument both force and velocity are under control; there is no error, as in efflux viscometers, due to contact of the liquid with the air or to cooling through evapn.; the high pressures possible adapt it for use on heavy liquids at room temp.; no corrections for sp. gr. are necessary because the level of the liquid in the vessel is above the orifice at the beginning of the test and below it at the end. By logarithmic plotting of time against pressure, readings on perfect fluids give straight-line curves, but colloidal solns. show deviations proportional to the degree of their colloidal character. A series of tests on *glycerol* indicates that internal stresses exist in this liquid so that it is not suited as a viscometer standard. The pressure required to drive an enamel through the instrument in 10 sec. is the numerical value of its brushing qualities. Tabulated readings and graphs are given. Discussion of the paper questions the effect of turbulent flow through the orifice at high pressures. A. de Waele prefers to plot pressures against vols. per sec. and then by extrapolation obtain "yield values," which are represented by the distance from the origin at which the curve intercepts the pressure axis. Yield values and rigidity, calcd. by a modified Poiseuille equation for plastics, Rigidity = (Pressure-Yield Value)  $\div$  Vol., on a number of liquids are given. F. A. WERTZ

The principles of application of paint and varnish. A. M. GIBBON. *J. Oil Color Chemists Assoc.* 3, 141-8(1920).—G. suggests a study of methods of application of paints and varnishes, and the manuf. of products particularly adapted to the specific

method of application. Spray coats and carelessly applied brush coats of paint are not as durable as carefully brushed out coats on account of inclusion of air in the film. The use of inert gas in the spray machine might increase durability of the film. Paints for spraying must be non-poisonous. Works of art on canvas are very permanent because the method of applying the paint precludes the inclusion of air. In bill poster printing, the pressure of the presses accomplished the same result. Tests on durability of paints applied to badly corroded Fe showed that the better the Fe was cleaned and scraped the longer the life of Pb paints, and that the reverse is true of Fe-oxide paints; hence the latter are better adapted for use by a careless painter. G. makes the "ridiculous" suggestions that paint chemists think about paints for use on creosoted timber; protective coatings sold in sheets and attached to surfaces by using pressure, etc.

F. A. WERTZ

**Grape-seed oil.** ELISABETH KLINGER. *Farben-Ztg.* 26, 6(1920).—Grape-seed oil on account of its partial soly. in alc. and its accredited low I no. of about 95, is usually grouped with castor oil as a non-drying oil; but in Spain it is used alone and mixed with linseed oil in paints. Investigation of a pure oil gave results which differ from those found in the literature: Sp. gr., 0.925; sapon. no., 192; I no., 130.9; its fatty acids, sapon. no. 199.5; I no. 133.5; m. p. 23–5°; congealing p., 18–20°. The raw oil dried to a rather sticky film in 4 days; with metallic driers it dried more rapidly to a sticky film. O absorption in 4 days was 7%, while a control test on linseed oil showed 14.7%. Mixts. of the oil with linseed oil dried to sticky films, but excellent results were obtained by heating 2 parts tung oil with 1 part grape-seed oil to 180° with 4% Pb resinate. On heating the oil to 300° it bodied much more rapidly than linseed oil, and in a few hrs. congealed to a gummy mass of linoxyn consistency.

F. A. WERTZ

**The present condition of the German pigment industry.** ANON. *Farben-Ztg.* 26, 2–3(1920).

F. A. WERTZ

**The condition of the (German) varnish industry.** ERICH STOCK. *Farben-Ztg.* 26, 3–4(1920).

F. A. WERTZ

**The French turpentine industry.** ANDRÉ JOLY. *J. Oil Color Chemists Assoc.* 3, 149–67(1920).—J. gives a detailed morphology of *Pinus maritima*, the French source of oleoresins; describes the method of extg. the resin; recovery of turpentine, rosin and by-products; and the compn. of pinene, and its reactions with  $H_2SO_4$ , HCl, NOCl, AcOH, BzOH.

F. A. WERTZ

**Use of the microscope in examining films.** HENRY A. GARDNER. *Paint Manufs. Assoc. of U. S., Circ.* No. 110, 6 pp., Nov. 1920.—A collection of photomicrographs at small magnification of the dried films of various varnishes, enamels, oils, and paints. The microscopic examn. of the films gives information in regard to their elasticity, porosity, hardness, resistance to  $H_2O$ , etc.

F. A. WERTZ

**Examination of a Mexican gum.** HENRY A. GARDNER AND ARTHUR REILLY. *Paint Manufs. Assoc. of U. S., Circ.* No. 11, 2 pp., Nov. 1920.—An unidentified gum, available in considerable quantity in Mexico, showed acid no. 90.8; sapon. no., 181.7; m. p., 80°; and consisted largely of small tear-like nodules of reddish brown color. The product appears to be a hardened exudation of some native shrub rather than a fossil resin, and its soly. in alc. suggests its use in spirit varnishes. Such a varnish dried to a hard, glossy, and more elastic film than shellac, but was very badly whitened by  $H_2O$ .

F. A. WERTZ

**The determination of benzene in turpentine.** A. ALLINA AND H. SALVATERRA. *Chem.-Ztg.* 44, 673–4, 697–8(1920).—In attempts to develop a method more satisfactory than that of Herzfeld (*Chem.-Ztg.* 33, 1081(1909); 34, 885(1910)) and of Marcusson (*Chem.-Ztg.* 33, 966, 978, 985(1909)) for the detn. of benzene in mixts. with turpentine or pine oil, the authors found (1) that the rise in temp. on addn. of  $H_2SO_4$  (2:1) furnishes

a rough test for the presence of benzene; (2) that the temp. coeff. of expansion of benzene is higher than that of turpentine or pine oil; but a method based on this fact is not adapted to a quant. detn.; (3) that a detn. of the amt. of Br absorbed, or of the increase in  $d$  on bromination is not reliable because the reaction does not proceed according to any definite rule; and (4) that a quant. detn. of the benzene content can be made by a steam distn. of the brominated oils. The latter test is carried out by dissolving 20 g.  $\text{KBrO}_3$  in 10 cc.  $\text{H}_2\text{O}$  and adding 20 cc. of the mixt. to be tested.  $\text{HBr}$ ,  $d$  1.49, is then added in 1 cc. portions to about 30% excess. The mixt. is allowed to stand for 1 hr. at  $40-50^\circ$ , when the excess Br and  $\text{HBr}$  are removed by  $\text{Na}_2\text{CO}_3$  and  $\text{As}_2\text{O}_3$ . After steam distn. the vol. of benzene is sometimes slightly contaminated by bromination products which can be removed by fuming  $\text{H}_2\text{SO}_4$ . F. A. WERTZ

**Varnish fume recovery.** CHESTER H. JONES. *Chem. Met. Eng.* 23, 771-6(1920).—A brief description, illust., of installations for the recovery of fumes in varnish manuf. The fumes from cooking gums and oils are passed through  $\text{H}_2\text{O}$  scrubbers which remove heavy particles, then through  $\text{Ca}(\text{OH})_2$  scrubbers, and finally pass into the air. Condensate from pipes and scrubbers is eventually returned to the kettles. The system for recovery of volatile thinners is so constructed that the condensed thinners flow back into the kettles; this reduces losses from 8% by the old method to 2% of the total thinners used, by the recovery method. J. reviews Gardner's report on fume losses (*C. A.* 14, 2094), and tabulates data from factory records on various varnishes. F. A. WERTZ

Lead (PULSIFER) 9. Resinous material from coal tar (U. S. pat. 1,355,103) 21.

AUSTERWEIL, G. AND ROTH, J.: *Gewinnung und Verarbeitung von Harz und Harzprodukten*. München und Berlin: R. Oldenbourg. M. 16.25.

EIBNER, ALEXANDER: *Die Oelfarbe in Kunst und Handwerk*. München: Verlag der Technischen Mitteilungen für Malerei. For review see *Farben-Ztg.* 26, 131(1920).

STOCK, ERICH: *Die Fabrikation der Spirituslacke*. Meissen: Verlag von Matthäus Bohlmann. 140 pp. M. 15. For review see *Farben-Ztg.* 26, 70 (1920) or *Chem. Umschau* 27, 200(1920).

**Paints.** DR. PLONNIL & Co. Brit. 147,799, July 9, 1920. Chalk is mixed with potash water glass which has been acted on by 5-10% of  $\text{KOH}$ .

**Paints; cements.** F. BENSA. Brit. 147,800, July 9, 1920. Pyrites ashes are freed from  $\text{H}_2\text{SO}_4$ , pulverized, freed from  $\text{H}_2\text{SiO}_3$  and ground with linseed oil to form a rust-preventing cement. A paint is obtained by dilg. the pasty cement with boiled linseed oil.

**Bituminous paint.** A. N. MCKAY AND W. L. WILLIS. U. S. 1,356,380, Oct. 19. A liquid mixt. suitable for coating boilers or smokestacks is formed by placing tar oil 500 gals. and "holder oil" 500 gals. in a cold still, adding aq. solns. of 25 lbs.  $\text{NaOH}$  and 12.5 lbs.  $\text{KMnO}_4$  and 12.5 lbs.  $\text{Ca}(\text{OH})_2$ , heating to about  $95^\circ$  for 6-8 hrs., drawing off a light distillate and then continuing the heating at a temp. of about  $65^\circ$  to drive off  $\text{H}_2\text{O}$  completely.

**Bituminous paint.** F. W. YEAGER. U. S. 1,355,102, Oct. 5. Cresols 1-3% are used in paints containing pitch dissolved in washed and refined tar distillates or condensates from illuminating gas, in order to prevent pptn. of insol. matter from the paint.

**Removing paint or varnish.** D. P. CLEVELAND. U. S. 1,355,074, Oct. 5. Paint and varnish are removed from the bodies of automobiles or other surfaces by the action of a jet of steam and  $\text{NaOH}$  soln. applied in such volume and at such a temp. as to disintegrate and flush off the substances of the coating.

**White lead.** W. P. THOMPSON. Brit. 147,752, July 8, 1920. White lead is prepd. by passing a current of air,  $\text{CO}_2$ , HOAc and  $\text{H}_2\text{O}$  vapors over unmelted anhydrous litharge kept constantly stirred. The  $\text{PbO}$  should be as free from metallic lead as possible, and may be prepd. in a similar manner to that referred to in 4,465 (1909). The mixing chamber consists of an horizontal cylinder fitted with a revolving shaft and blades and is supplied with  $\text{PbO}$  from a feed-pipe. Air and  $\text{CO}_2$  are passed into the heating chamber and become mixed with HOAc and  $\text{H}_2\text{O}$  vapors from the dil. HOAc contained therein. By regulating a burner the temp. of the gaseous current leaving by the pipe is kept within the limits  $26-60^\circ$ , whereby the formation of crystalline white lead of low covering power, due to the presence of too much moisture, is prevented. Unabsorbed gases escape by a pipe and the white lead is drawn off periodically through a shutter. Cf. C. A. 13, 1645.

**Polishing composition.** E. A. LEAKE. U. S. 1,356,075, Oct. 19. A mixt. suitable for polishing varnished woodwork is formed of linseed oil 22.7,  $\text{HCl}$  1.5, alc. 5,  $\text{SbCl}_3$  4.5, HOAc 62, methyl orange 0.8 and oil of mirbane 3.5 parts.

**Lithopone.** J. L. MITCHELL. U. S. 1,356,387, Oct. 19. In the manuf. of lithopone, a product composed of  $\text{BaSO}_4$  and  $\text{ZnS}$  is washed, dried and pulverized, calcined in a muffle furnace at  $700-950^\circ$  while agitated, quenched in cold  $\text{H}_2\text{O}$  and then dried.

**Solvent for pyroxylin.** E. M. FLAHERTY. U. S. 1,356,440, Oct. 19. A solvent for pyroxylin adapted for making *lacquers* or *varnishes* is formed of normal butyl acetate 40% mixed with 60% of  $\text{C}_6\text{H}_6$  or benzene or normal butyl acetate 41,  $\text{C}_6\text{H}_6$  or benzene 50 and normal butyl alc. 9 parts. Cf. C. A. 14, 2263.

**Preparing metal surfaces, especially of magnesium and its alloys, for coatings of varnish and the like.** F. MEYER. Ger. 317,656, May 3, 1919. The surface of the metal is treated with drops of acid or the like to produce a porous surface. The article is then cleansed mechanically or chemically.

**Synthetic resins.** H. BUCHERER. Brit. 148,139, July 9, 1920. The free phenolic groups of the resinous condensation products from phenols and  $\text{HCHO}$  are substituted by org. radicals, such as alkyl, aralkyl, or acyl groups, whereby the products become insol. in alkali and their soly. in org. solvents is modified. Examples are given of the prepn. of *p*-toluenesulfonic, acetic and benzoic esters by means of  $\text{MeC}_6\text{H}_4\text{SO}_2\text{Cl}$ ,  $\text{AcCl}$  and  $\text{BzCl}$ , resp., a benzyl ether by means of benzyl chloride, a carbonic ester by means of  $\text{COCl}_2$ , and mixed ether-esters by partial etherification by benzyl chloride, followed by esterification by benzoyl or acetyl chloride or  $\text{COCl}_2$ . The *p*-toluenesulfonic, benzoic, and acetic esters, the benzyl ethers, and the mixed ether-esters are sol. in various organic solvents; the carbonic esters are, however, practically insol. and of high m. p.; they may be prepd. *in situ* upon textiles, wood, paper, etc., by treating with  $\text{COCl}_2$  the material impregnated with alk. solns. of the resins.

## 27—FATS, FATTY OILS AND SOAPS

E. SCHERUBEL

**The glycerides of fats and oils. IX. The glycerides of coconut oil.** A. BÖMER and J. BAUMANN. Münster. Z. Nahr. Genussm. 40, 97-151 (1920); cf. C. A. 8, 1174, 2433; Z. Nahr.-Genussm. 28, 586. Caprylic acid was found to be present in coconut oil but no caproic or capric acid could be detected. Palmitic and stearic acids as well as oleic acid were found. The glycerides of the satd. fatty acids consist chiefly of a caprylolauromyristin (m.  $15.0^\circ$ ), and a myristodilaurin (m.  $33.0^\circ$ ), in addition to a laurodimyristin (m.  $38.1^\circ$ ). Both of the most insol. glycerides of coconut oil, palmitodimyristin (m.  $45.1^\circ$ ), and a stearodipalmitin (m. p. of the impure glyceride  $55.0^\circ$ ) occur only in small amts. in coconut oil.

L. D. ELLIOTT



A new catalyzer for hydrogenating fats. G. GROTH. *Seifensieder-Ztg.* 47, 713-4 (1920).—This new catalyzer is made by pptg. a soln. of a Ni and Mg or Al salt by a soln. of  $\text{Na}_2\text{Si}_2\text{O}_5$ , washing and drying the ppt. and reducing same in a current of H. A dark gray powder results which is preserved in oil. By using this on the basis of 0.2 to 0.5% Ni a m. p. of 42 to 48° can be obtained in 15 or 20 min. at a temp. of 160° with edible fats. Three tables are given showing comparison with other catalyzers.

E. SCHERUBEL

The use of sulfuroil in textile and household soaps. M. N. *Seifensieder-Ztg.* 47, 711-2 (1920).—Sulfuroil is a product which is obtained from the residue of olive oil manuf. by a special process. The oil contains about 75% free fatty acids and is of different consistencies and always makes a green soap. It is characteristic of this soap that it takes about twice as long for it to solidify as other soap. By mixing sulfuroil with other fats harder soaps can be made which are capable of being filled and making satisfactory household soaps. The straight sulfuroil soap is especially suitable for the textile industry.

E. SCHERUBEL

Note on the chemical composition of two palm kernels from Madagascar. G. CLOT. *Mat. grasses* 12, 5661-2 (1920).—The 2 seeds analyzed were *Hyphaene shaton* and *Borassus madagascariensis*. The former contd. moisture 12.10%, fat 8.08%, N 5.95%, sol. ash 1.01%, insol. ash 1.38%, celluloses 71.48%. The latter contd. moisture 10.25%, fat 0.51%, N 4.84%, sol. ash 1.20%, insol. ash 0.54%, cellulose 82.66%. The I no. of the oil from hyphaene was 21.9 and the sapon. no. 245.3. An hydrolysis of the cellulose from *B. madagascariensis* gave 10.91 g. mannose and 2.64 g. glucose from 80 g., while 10 g. of hydrolyzed cellulose from hyphaene gave 2.47 g. mannose and 0.965 g. glucose.

E. SCHERUBEL

The seed oil of the "condori" tree (*Adenanthera pavonina* L.). A. DIEDRICHS AND L. KNÖRR. *Neuss. Z. Nahr. Genussm.* 40, 153-5 (1920).—A description of the seed and table of consts. of the oil are given.

L. D. ELLIOTT

Sediments in vegetable oil deodorizers. J. MARTIN. *Mat. grasses* 12, 5635-6 (1920).—Oils deodorized at a temp. of 300-350° often gave a gelatinous deposit on the sides of the app. The sediment from a palm oil refined with lime and  $\text{Na}_2\text{Si}_2\text{O}_5$  gave: Moisture, 8.3%, neutral oil 30.1%, free fatty acids 3.3%, iron soap 12.5%, org. matter 19.5%, Fe oxide and silicate 17.7%, lime 5%, undetd. mineral matter 3.6%.

E. SCHERUBEL

Determination of ammonia in oil and fat preparations. N. WELWART. *Chem.-Ztg.* 44, 719 (1920).—Turkey red oils and various textile oils are prepd. with  $\text{NH}_3$  and in detg. this the total fat is washed with water contg. a small amt. of  $\text{H}_2\text{SO}_4$ . The combined washings are distd. with NaOH and the  $\text{NH}_3$  caught in a standard acid soln. This method is not always suitable and the following one is suggested: A weighed amt. of the sample is dild. with water in an Erlenmeyer flask and some pumice and an excess of  $\text{CaCl}_2$  added together with NaOH soln. The  $\text{NH}_3$  is distd. into acid and titrated.

A. SCHERUBEL

The possibility of substituting inorganic colloids for soap. G. WEISSENBERGER. *Tech. Hochschule, Wien. Kolloid.-Z.* 27, 69-78 (1920).—The shortage during the war led to the search for a substitute for soap. The detergent action of soap depends largely upon the fact that as an emulsoid colloid it emulsifies fats, adsorbs dirt particles, and lowers the surface tension of water so that the fabric is more easily wetted. Frothing facilitates removal of dirt in the drain water, but is not essential. Any emulsoid of the same degree of dispersion should have similar detergent properties. As substitutes the inorg. colloids are most desirable because of cheapness. The natives of Algiers and Tunis use a natural mineral for washing. This is an alkali and earth metal silicate containing free gelatinous silica, clay, and alkali carbonate. A larger number

of clays from Austria-Hungary were studied as possible soap-like colloids. As criteria of the detergent action were considered (1) the ability to absorb water, (2) the Spring effect (*C. A.* 3, 1599, 1613), (3) the formation of highly viscous solns. and finally jellies on evapn. the suspensions. No clay passed these tests as none had a sufficiently high degree of dispersion. Attempts to increase the dispersity by mechanical grinding or by treatment with acids and bases failed. The clay from Gaura, Siebenbürgen contained 40% of a substance obtainable by sedimentation, which was probably an adsorption complex of gelatinous silica and smaller amts. of  $\text{Al}(\text{OH})_3$ , and gave stable suspensions of a high degree of dispersion. Evapn. gave a reversible jelly. The Spring effect was positive. In practical washing tests, both on a small scale and in a steam laundry, it proved useful. Attempts to produce a froth by adding small amts. of soap failed because the colloid adsorbed the soap.

F. L. BROWNE

"Babasso" nuts and their oil (DIEDRICHS, KNÖRR) 12. Fatty acids from hydrocarbons (Brit. pat. 147,905) 22.

**Lanolin.** W. C. BROADGATE. U. S. 1,354,376, Sept. 28. Wool grease is heated sufficiently to convert it into a thin even mixt. and an alkali metal peroxide is added as a bleaching agent. The mixt. is then agitated, allowed to stand for completion of the bleaching action and then centrifuged to sep. the lanolin from the remainder of the mass.

**Extracting fats, etc., from bones.** A. FAITLOWITZ. Brit. 148,161, July 9, 1920. The residual fat and albumin are extd. from bones that have already been treated in an autoclave for extg. fat, by boiling the crushed bones with  $\text{H}_2\text{O}$  and  $\text{Na}_2\text{CO}_3$ . The fat is skimmed from the settled liquid, and the soln. of albumin is withdrawn from the  $\text{CaO}$ , acidified with  $\text{HCl}$ , mixed with 10% of  $\text{NaCl}$ , and treated with a current of  $\text{CO}_2$  to ppt. the albuminous matter. The albuminous matter is used in the manuf. of meat exts.

**Catalyst; hydrogenating fats and oils.** C. & G. MÜLLER SPETSEFETTFABRIK AKT.-GES. Brit. 148,111, July 9, 1920. Non-pyrophoric metal catalysts adapted for use in the hydrogenation of fats and oils, are prepd. by pptg. a soln. of a catalytic metal with a soln. containing a usual precipitant such as  $\text{Na}_2\text{CO}_3$  or caustic alkali, together with borax, and then reducing the ppt. in a current of  $\text{H}_2$ . In examples, a soln. of  $\text{NiCl}_2$  is pptd. with a soln. containing  $\text{Na}_2\text{CO}_3$  and borax so as to give a ppt. of  $\text{Ni}$  carbonate and borate, which is afterwards dried, heated to decompose the carbonate, and then reduced in  $\text{H}_2$ ; a soln. of  $\text{NiO}$  dissolved in  $\text{HCl}$  is mixed with kieselguhr, and pptd. with a soln. containing  $\text{Na}_2\text{CO}_3$  and borax, and the ppt. then reduced as usual; a cobalt salt soln. is mixed with kieselguhr and pptd. with a soln. containing a usual pptg. agent and borax and the ppt. is washed, dried, and reduced in  $\text{H}_2$ .

**Regenerated exhausted catalyzers for fat hardening.** N. GOSLINGS. Norw. 30,587, Apr. 6, 1920. The exhausted catalyzer is boiled with dil. alc. or dil. alkali soln. without previous removal of the organic residue, thoroughly washed and then reduced in a current of  $\text{H}_2$  with gradual increase of the temp. to at most  $650^\circ$ , until the org. matter is completely burned out.

**Extracting oils.** G. D. ROGERS. Brit. 147,834, July 9, 1920. See U. S. 1,326,968 (*C. A.* 14, 855).

**Extracting oils.** M. WILBUSCHEWITSCH. Brit. 147,745, July 8, 1920. In an app. for the continuous extn. of oil from seeds, etc., by treatment on the counter-current principle with solvent and removal of the ext. by pressure, the seed passes through a funnel and pipe to the bottom of a solvent or ext. container from which it is carried through solvent by conveyors to a press. A suitable app. is specified.

## 28—SUGAR, STARCH AND GUMS

F. W. ZERBAN

The sugar industry of France since the war. T. H. MURPHY. *J. Ind. Eng. Chem.* 12, 1124-6(1920), illus. E. J. C.

Formula for Steffen extraction. WALLACE MONTGOMERY. *Sugar* 22, 576(1920).—An empirical method for calcg. probable results of com. process as based on lab. data, showing sugar extn. and molasses accumulations. C. H. CHRISTMAN

The clarification of cane juice without chemical treatment. F. W. ZERBAN. *Louisiana Planter* 65, 204-6, 220-2(1920).—See *C. A.* 14, 2868. C. H. C.

Recent advances in sugar clarification. W. D. HORNE. *Louisiana Planter* 65, 175(1920).—The use of the Dorr clarifier and the Williamson clarifier provides continuous clarification of raw juices and raw sugar solns. Greater economy and better sugars result from the use of these devices. C. H. CHRISTMAN

Experiments concerning the inversion constant of pure cane sugar. OTTO SCHREFFELD. *Z. Ver. Zuckerind.* 70, 402-8(1920).—In a redetn. of this constant, carried out in 1910, using Herzfeld's method, the Clerget factor at 20° was found to be  $\approx 133$ . The lower figure, 132.66, established in 1888, was probably due to small amts. of raffinose in the sugar used. The factor is a linear function of the concn. of sucrose, increasing with the latter from 132 for 4 g. per 100 cc., to 133 for 13 g. in 100 cc. The temp. of 67-70° prescribed by Herzfeld is apt to cause decompn. of the invert sugar; this danger can be avoided by inverting at 60°, but then the time of heating must be increased to 10 min. (cf. *C. A.* 14, 2561). Inversion carried out at 19° is complete after 48 hrs., but not after 24 hrs. It makes no difference whether at first the sugar is dissolved with 75 cc. of water, or with as little as 60 cc. In the analysis of low-grade products the amt. of HCl must be increased and the time of heating lengthened.

F. W. ZERBAN

Changes in the polarizing constants of sugars during refining. A. F. BLAKE. *J. Ind. Eng. Chem.* 12, 1014-7(1920).—B. has detd. the ratio: [Sucrose-Polarization]/Invert Sugar throughout the bone black refining process. It was found that the ratio varies in different raw sugars, but averages about 0.3 which means equal quantities of dextrose and levulose. During the refining process this ratio decreases, being below 0.1 in the soft sugars and barrel sirup. The decrease of the ratio occurs (1) in the defecation of scum and char waters, centrifugal washings, and press waters, where the lime added destroys more levulose than dextrose; (2) in the char filtration, the bone black adsorbing more levulose than dextrose. Other changes appear to take place in the char filters, but they have not been explained as yet. Attention is called to the fact deduced from these results that the actual sugar losses in process are greater than appears from the direct polarizations, and that the actual amt. of organic non-sugars removed is less than that calcd. on the same basis.

F. W. ZERBAN

The removal of calcium salts from carbonatated juices. VL. STANEK. *Listy Cukrovar.* 38, 21-3; 29-33(1919).— $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{SO}_3$  are used as pptg. agents. The pptn. of lime, using soda in conjunction with carbonatation, is more complete at the temp. of boiling than at around 70°. It is also more complete if the soda is added before the 2nd, than if it is added after the final carbonatation. Carbonatation with the addition of lime removes more lime salts than simple carbonatation. This is true even when no soda is added if the juice contains sufficient natural alkalinity. The combined action of lime, carbonatation, and of natural or added alk. carbonates at boiling temp., removes the lime salts until their solubility limits are reached. This removal can go to practical completeness if a certain excess of alk. is present.  $\text{Na}_2\text{SO}_3$

can be used just as effectively as  $\text{Na}_2\text{CO}_3$ , the former having a mild decolorizing action. Molecular quantities of soda will not ppt. the lime quantitatively even if carried out at boiling temp. If the pptn. is made with an insufficient amt. of soda then, to obtain the greatest effect, carbonatation at boiling temp. with the addition of lime is used.

JOHN M. KRNO

**Gypsum in the juices and in the carbonatation sludge.** VL. STANĚK. *Z. Zuckerind. gechoslov. Rep.* 44, 69-72(1919); *Listy Cukrovar.* 38, 45-8(1919).—Analysis showed that there is a great deal more  $\text{CaSO}_4$  in the carbonatation sludge than one would expect to find, judging from its soly. in sugar juices. Investigation of the conditions for pptg. gypsum and the nature of the ppt. obtained, disclosed the following:  $\text{CaSO}_4$  can be leached out from the sludge very slowly and then only incompletely. Soln. of sulfates is obtained more readily by supersatn. with  $\text{CO}_2$ .  $\text{CaSO}_4$  is pptd. only in traces, if at all, by  $\text{Ca}(\text{OH})_2$ .  $\text{CaCO}_3$ , when freshly formed by the carbonatation of  $\text{Ca}(\text{OH})_2$ , carries with it  $\text{CaSO}_4$  from the soln. The larger the amt. of lime that is carbonated, the greater will be the amt. of gypsum pptd. The alkali sulfates are partly causticized and the  $\text{H}_2\text{SO}_4$  tends to go into the carbonatation sludge, when  $\text{Ca}(\text{OH})_2$  is carbonated in their solution. Here again, the larger the amt. of lime that is carbonated, the greater will be the amt. of gypsum pptd. The pptn. is more complete if the juice is satd. to an alkalinity of only 0.1% of  $\text{CaO}$ , for if oversatd. the gypsum is again decomposed by the alkali carbonate. Upon these results S. bases his opinion that the soda used for deliming juices, can be replaced less expensively by Glauber's salt and even by  $\text{NaHSO}_4$ .

JOHN M. KRNO

**The ammoniacal frothing of after-product massecuites.** KAREL URBAN. *Z. Zuckerind. gechoslov. Rep.* 44, 21-3(1919); *Listy Cukrovar.* 38, 37-9(1919).—U. mentions a case of frothing in massecuites occurring in the coolers at a temp. as low as 70-75°. The rise in vol. equalled 5-7%.  $\text{NH}_3$  was constantly given off but the alkalinity of the massecuite did not drop. This occurrence was especially noticed when working with juices from partly frozen beets. U. expresses the opinion, from analytical results obtained, that this phenomenon is due to the action of sugars on amino acids, producing  $\text{CO}_2$ , and to the evolution of  $\text{NH}_3$  from the decompn. of  $\text{NH}_4$  salts of amino acids when the juices are coned.

JOHN M. KRNO

**Microscopical grain in molasses.** JAR. DĚDEK. *Listy Cukrovar.* 38, 264-8(1920); *Z. Zuckerind. gechoslov. Rep.* 45, 1-7(1920).—The method for the detn. of microscopical grain in molasses as described by Kalshoven (*C. A.* 14, 860), when applied in the investigation of the high purities of beet molasses during the last campaign, was found to give unreliable results. This unreliability was caused chiefly by the contraction in vol. of the molasses due to the diln. and subsequent heating of the sample, in order to bring about the complete soln. of the microscopical grain. To avoid this error, the molasses was placed in a small autoclave of special design which was then immersed in a glycerin bath at 110° for 15 min., agitating constantly. After cooling the refractometer reading was obtained. From the difference between this and the original refractometer reading, the % of grain in suspension was calcd. By a very efficient arrangement of illumination the accuracy of the reading was doubled. The method as described gave lower results than the original Kalshoven method. At a concn. of 70-85° Brix the accuracy decreased to 1/2. At 95° Brix it was only 1/3, showing that the accuracy decreased rapidly with the increase in the total dry substance.

JOHN M. KRNO

**The treatment of after-products according to Dr. Claassen.** CHRIST. MRASEK. *Listy Cukrovar.* 38, 97-9, 105-6(1920); *Z. Zuckerind. gechoslov. Rep.* 44, 129-34(1920).—A review and discussion of Claassen's work on the crystn. of after-products.

JOHN M. KRNO

**Pumps, centrifugals and other rotating machinery used in sugar refineries.** K. ZIALECKI. *Listy Cukrowar.* 38, 1-3, 5-7, 16-7, 23-5, 33-5(1919).—The author deals mainly with an extensive description of different types of pumps and centrifugals used throughout the sugar refinery.

JOHN M. KRNO

**Molasses as fuel and the manufacture of potash from the ashes.** G. E. G. VON STIETZ. *Louisiana Planter* 64, 348-50(1920).—See *C. A.* 14, 2427.

C. H. CHRISTMAN

**Maple sugar sand.** J. F. SNELL. *Proc. 27th Ann. Meet. Vt. Maple Sugar Makers' Assoc.* 38-44, (1920).—The use of maple sugar sand as a source of malic acid or malic acid salts is discussed. From 50 to 160 tons of acid are available from this source which at present appears to be the most economical. It is necessary in its prepn. to wash out the sugar, dry promptly and keep dry.

H. A. LEPPER

**Levulose sirup.** J. J. WILLAMAN. *Univ. Minn. Science* 52, 351-2(1920).—W. proposes the production of levulose, the sweetest known sugar, in sirup form from the Jerusalem artichoke which gives large yields per acre and contains 12-14% of inulin. Methods for large scale manuf. are yet to be worked out.

F. W. ZERBAN

**Fundamental methods in the analytical chemistry of sugar.** VI. EMILE SAILLARD. *Mon. sci.* [5] 10, 97-101(1920); cf. *C. A.* 14, 471.—Of the various apparatus on the market for prep. beet pulp to be used in the analysis by the cold aqueous digestion method, S. prefers the Herles press. It does very good work and the maintenance cost is low. The "Sans Pareille" press also gives good results when new and well constructed, but it breaks easily. In the methods based on constant vol. the air or gas bubbles enclosed in the pulp are liable to cause error, and they must be carefully removed by agitating the flask and by adding alc. or ether. There is a further cause of error due to variation in the marc content of the beets. This error may, for the German normal wt., be as high as 0.17% of sucrose in the constant wt. methods, and 0.04% in the constant vol. methods; with the double French normal wt. they may reach 0.11 and 0.02%, resp.

F. W. ZERBAN

**A cryoscopic method for the estimation of sucrose.** HENRY H. DIXON AND T. G. MASON. *Louisiana Planter* 64, 397-8(1920).—See *C. A.* 14, 2498.

C. H. C.

**The analysis of massecuite by means of the refractometer.** STEGFRIED LAURENS MALOWAN. *Z. Zuckerind. czechoslov. Rep.* 44, 392(1920).—The methods in use for the detn. of dry substance and purity in factory sugar liquors require sep. weighed samples for each detn. The one exception is the diln. method of Koydl in which both the polarization and the refractometer reading are obtained on the same sample. The author proposes the following method as more accurate and rapid: 26.05 g. are weighed into a tared beaker and water is added until the mixture weighs 100 g. A few drops of this soln. are taken and  $n_d$  detd. in a Zeiss refractometer. The soln. is then transferred to a 100 cc. flask, rinsing the beaker with a sufficient quantity of lead acetate soln. After filling to mark, the ppt. is filtered off and the filtrate polarized. Two tables are given, one showing the agreement in the results obtained by the foregoing and Koydl's diln. methods, the other, giving the % of dry substance for each corresponding refractometer reading.

JOHN M. KRNO

**The gum disease of sugar cane (WILBRINK) 11D. The transition to lignite firing in starch factories (GESBLL) 21. Generator gas from bagasse (HATTINK) 21. Motor alcohol from molasses (CHILD) 16.**

**Methods of Analysis and Laboratory Control of the Great Western Sugar Company by the Chemical Dept. of the Great Western Sugar Co.** 1st Ed. Denver: The Great Western Sugar Co. 259 pp.

**Protein and phosphates from corn steep acid waste waters.** A. GIESCKE. U. S. 1,354,822, Oct. 5. In starch manuf. where the corn has been steeped in  $\text{SO}_2$  soln., the acid waste water is partially neutralized with dry  $\text{Na}_2\text{CO}_3$  and the ppt. formed is mixed with the solids of table tailings to form a compd. gluten meal.

## 29—LEATHER AND GLUE

ALLEN ROGERS

**The determination of water-soluble in leather, 1920 committee report.** G. L. TERRASSE, *et al.* *Am. Leather Chem. Assoc.* 15, 581-92(1920).—The purpose of the committee was to ascertain the best conditions for the determination of the figure called "water-soluble." Portions of a uniform, finely ground sample, with instructions, were sent to each member of the committee. Figures of the various members were concordant in each case. The following recommendations as a basis for the detg. of "water-sol." in leather were made; the sample must be finely ground and free from grease; the extn. must be carried out in the Reed-Churchill extractor; 30 g. of leather shall be used for each extn.; the temp. of extn. shall be  $50^\circ$ ; 2 l. of percolate shall be collected; there shall be no digestion period and the time of extn. shall be 3 hrs. Brief discussion followed.

C. F. JAMESON

**Electrochemistry of tanning materials. III. Electroösmosis of mixtures, adsorption compounds, and split products, of tanning materials.** GEORG GRASSER. *Collegium* 602, 277-90(1920); cf. *C. A.* 11, 1920. Cf. *C. A.*—*Quebracho and mangrove*: The electroösmosis of this mixt. gave rise to a blood-red coloration in the cathodic dialyzate in the first step of the expt. Pure quebracho gives a brown color; sulfite quebracho ext., no color. After 15 min. of action by the elec. current, the cathodic dialyzate of pure quebracho ext. yields at most a pink coloration, which remains unchanged on the addition of  $\text{KNO}_3$ . In the presence of mangrove a blood-red color appears in the cathodic dialyzate, which changes to a raspberry color on the addition of  $\text{KNO}_3$ . Addition of  $\text{NaOH}$  is beneficial, since in its presence the cathodic dialyzate of pure quebracho ext. remains colorless even after 1 hr.'s action by the current, whereas in the presence of mangrove the blood-red coloration appears in 15 min. This makes possible the detection of mangrove adulterations of quebracho exts. with greater certainty than do the methods heretofore employed. *Quebracho and chestnut*: In the anodic dialyzate of the first two steps of the expt. there appeared a tanning material giving a dark color with the ferric ion, and a slight reaction with  $\text{KNO}_3$  and  $\text{Na}_2\text{SO}_3$ , indicating the absence of chestnut ext. and the anodic migration of the quebracho tanning material only. In the dialyzate of the next two steps, the anodic migration of the tanning materials of both became evident. *Quebracho and sulfite cellulose extract*: The ligninsulfonic acid of this mixt. migrates both anodically and cathodically, whereas the Na salts migrate anodically. *Quebracho and neradol D*: Due to the salts present in the neradol as impurities, the process of electroösmosis was slow as compared with that of pure quebracho ext. In the treated residue there appeared but a slight ppt., the fluid remaining clear, and giving no fuchsin-red color with concd.  $\text{H}_2\text{SO}_4$ . It would seem that all the quebracho ext. migrated completely or partly turned into insol. compds., and that neradol D alone was present in the residual fluid. The scanty reaction of the residue with  $\text{H}_2\text{SO}_4$  and the formation of a ppt. in the anodic dialyzate on the addition of  $\text{BaCl}_2$ , permit to establish the presence of neradol D in quebracho by the electroösmotic process. *Quebracho and sodium sulfite*: No cathodic migration of tanning material at first took place. but the anodic migration of the  $\text{SO}_4$  ions occurred even in the first stage of the expt. In the later stages the anodic migration of the tanning material became distinctly evident. The examn. of the treated residue indicated that the pres-

ence of the Na salt facilitated only slightly the decompn. of the quebracho tanning materials by the elec. current. *Quebracho, sodium sulfite and sodium bisulfite*: Generally speaking, the electroosmotic process ran along the same as in the above expt. The  $\text{SO}_3$  ions, however, appeared in the cathodic dialyzate. The treated residue was turbid, and at the end showed the presence of a ppt. *Quebracho and dextrose*: The detn. of the presence of sugar in tanning materials and in leather exts. by means of *o*-nitrophenylpropionic acid presents difficulties in those cases where the sugar is present in small quantities and where the addition of NaOH interferes with the rapid and complete sepn. of the reaction compds. in  $\text{CHCl}_3$ . Electroosmotic treatment of such sugar-containing exts. gave light brown dialyzates, which became distinctly bluish green on boiling in the presence of NaOH and *o*-nitrophenylpropionic acid. Subsequent shaking with  $\text{CHCl}_3$  led to the rapid sepn. of indigo blue and the formation of a deep blue soln. The sugar migrated both, anodically and cathodically. The migration, however, was slow, and the treated residue still contained considerable amts. of the sugar. Nevertheless, this process of sugar detn. has advantages over the old methods and should be used as conclusive evidence where the old methods yield questionable results. *Quebracho and auramin*: (Addition of 0.5% auramin acidified with AcOH). Electroosmosis of this mixt. failed to establish the presence of the dyes in the mixt., probably because of the complete decompn. by the elec. current. *Quebracho and albumin*: Spent quebracho liquor was used which was of a slightly acid reaction, contained a trace of tanning materials, various split products, albuminous substances, and considerable quantities of Ca salts. The process of electroosmosis proceeded normally, the anodic dialyzate containing the tanning materials and the cathodic albuminous substances. The treated residue showed no traces of either. Purification of tanning materials according to the above expt. by first pptg. them with gelatin, dissolving in an alk. soln. and treating electrolytically is not very promising, because of the changes in the tanning materials due to the secondary processes arising during the electroosmosis. However, a more thorough study should be made of such a possibility. *Pb compds. of quebracho tanning materials*: Filtered quebracho ext. was treated with Pb acetate and the ppt. redissolved in warm AcOH. In the first stages of the electroosmotic process, the anodic dialyzate was strongly acid and practically free from tanning materials. Later, perceptible amts. of unchanged tanning materials appeared. The treated residue was free from Pb, and though of dark color, gave only very slight reactions characteristic of quebracho tanning materials. The sepn. of the constituents of lead tanning compds. seems to be thoroughly accomplished by the process of electroosmosis, but this method is probably of no value for practical purposes. B. S. LEVINE

Some problems in the study of tanning chemistry. E. STIASNY. *Collegium* 1920, 225-66.—A lecture delivered before the Ass'n of German Chemists, Eastern section, Darmstadt. B. S. L.

**Aldehyde tannage.** W. MOELLER. *Collegium* 1920, 185-99.—As a result of a long series of expts. it is concluded, contrary to the investigations of Gerngross, that: In the first stage of aldehyde tannage, the aldehyde enters into chem. reaction with the split products of the hide. These reaction products do not serve to tan the hide nor to make it water-insoluble. The fact that the protein split products do not harden when acted upon by  $\text{HCHO}$ , but form water-sol. products, is proved by the expts. of Mavrojanis and by the formal titration of Sørensen. Tanning factors here, more than in any other tanning process, are, in accordance with M.'s peptization theory, the polymers and the condensation products built up through the hide by the excess of  $\text{HCHO}$  and by fermentation. B. S. L.

**Swelling and falling of white hide in vegetable tan liquors.** GEO. D. McLAUGHLIN AND RALPH E. PORTER. *J. Am. Leather Chem. Assoc.* 15, 557-81(1920).—Writers

state that official method of analysis of tanning material or a tannery liquor does not state value of samples in regard to the character of tannin, or depleting power of the various constituents. They show some of the actions upon white hide of each of the main ingredients in some of the vegetable tanning materials and their inter-relation. They also outline methods for a broader assay of tanning materials and conditions. A certain tail liquor caused white hide to lose 3% of its wt. after 24 hrs. immersion. It contained lactic, acetic, gallic and pyrogalllic acids. Lactic acid has great swelling power and a mellowing effect on astringent liquors. With chestnut and quebracho fresh liquor it does not darken the color or ppt. the liquor unless high concns. are exceeded.  $\text{CH}_3\text{COOH}$  has little or no swelling power, great mellowing power, and represses swelling power of lactic acid. It increases the depleting power of gallic acid. Gallic acid has great depleting power, represses lactic acid and increases action of  $\text{CH}_3\text{COOH}$ . It has mellowing properties. Pyrogalllic acid has properties similar to gallic. Lactate and acetate of Ca have depleting and mellowing power. Complete tables and graphs showing the effects of varying concns. of the different acids upon white hide under different conditions are given. The work is interpreted practically and the adoption of a more satisfactory official method of analysis is urged.

C. F. JAMESON

Order of diffusion of tanning extracts through gelatin jelly and their relation to the results obtained by Wilson and Kern. ARTHUR W. THOMAS. *J. Am. Leather Chem. Assoc.* 15, 593-5(1920).—W. and K. have shown that exts. containing greatest amts. of non-tans give largest errors by the official method. They have given a list of exts. in order of percentage error. T. allowed 1% solns. of various exts. to diffuse through jellies (5% gelatin in water) containing 0.1%  $\text{FeCl}_3$ . At intervals the diffusions as shown by the black zones were measured. With the exception of osage orange the order was similar to order given by W. and K. The discrepancy of osage orange is attributed to its low content of non-tans, which give black coloration with ferric iron. T. considers similarity of order very significant.

C. F. JAMESON

Some considerations on the testing of dyes in the leather industry. LOUIS J. MATOS. *J. Am. Leather Chem. Assoc.* 15, 553-7(1920).—Lab. tests of dyes on leather are now far from satisfactory. Suggestions for improvement are given. Before dyeing, chrome leather should be very carefully neutralized. The period between tanning and dyeing should be short. Advice on the application of acid and basic colors is given. Careful selections of samples (3" × 6" from various parts of the hide), removal of all grease and dirt, and standardization wherever possible are emphasized. App. for tests imitating tannery practice is described and recommended.

C. F. JAMESON

The mechanism of bating. JOHN ARTHUR WILSON. *J. Ind. Eng. Chem.* 12, 1087-90(1920).—The mechanism of the process of bating unhaird skins prior to tanning is shown to consist of two distinct parts: (1) reducing the skins to a condition of minimum swelling; (2) digesting the elastin fibers present in outer layers of the skins. The second action is shown very clearly by means of photomicrographs of cross sections of skin bated with trypsin for different lengths of time. Also in *J. Am. Leather Chem. Assoc.* 15, 649-54(1920).

J. A. WILSON

Disinfection of the skin of glanders-infected animals with milk of lime. LUDWIG PREIFFER. *Arch. wiss. prakt. Tierheilk.* 46, 62-86(1920).—Pathological and bacteriological study of the efficacy of milk of lime as an external disinfectant.

H. F. ZOLLER

The Miles acid process on tannery waste (DORR) 14. Tannins. V. Phloroglucinol tannins and catechols (FREUDENBERG) 10. Grasselli Medal award (ANON.) 13. Leather substitutes (Brit. pat. 147,910) 30.



## 30—RUBBER AND ALLIED SUBSTANCES

JOHN B. TUTTLE

Cultivation and exploitation of castilloa. E. DE WILDEMAN. *Caoutchouc & gutta-percha* 17, 10431-2(1920).—A review. G. S. W.

The seeds of Hevea and the conditions of their germination. E. DE WILDEMAN. *Caoutchouc & gutta-percha* 17, 10429-31(1920).—A review, describing the flowers and seeds of *Hevea brasiliensis*. G. S. W.

Further data on the influence of tapping on latex and rubber. O. DE VRIES. *Arch. Rubbercult.* 4, 313-34(1920).—(a) Prolongation of the tapping cut, (b) increase in the number of tapping cuts on one tapping surface, (c) exceptionally drastic tapping (to the wood), are found to produce in the latex and rubber the same effects (one of the most noticeable of which is a rise in the rate of cure) as heavier tapping in general (cf. *C. A.* 12, 779, 1135; 13, 386, 1775). No differences in vulcanizing properties were to be observed (a) between rubber from low and from high cuts; (b) between rubber from an earlier and from a later tapping, in the case of trees tapped twice a day; (c) between rubber from trees tapped early and trees tapped later in the day, in the case of trees tapped once daily. G. S. WHITBY

Bacteriological study of the coagulation of Hevea latex. DENIER AND G. VERNET. *Caoutchouc & gutta-percha* 17, 10491-3(1920); cf. *C. A.* 11, 2971.—In the bacteriol. examn. of 6 samples of latex at Saigon 1 anaerobe and 27 aerobes or facultative aerobes all of which are described, were found. Only one bacterium (belonging to the latter group) was found in all samples; and this was the only one which attacked the "sugar" of Hevea latex (called by the authors "dambosite"), producing acid. G. S. WHITBY

The aging of certain rubber compounds. I. R. RUBY AND HARLAN A. DEPEW. *J. Ind. Eng. Chem.* 12, 1155-6(1920).—Using the method of Geer, 3 compds. were tested, the oven temp. being 70°. The compd. containing lithopone ages less satisfactorily than that containing only zinc oxide for a filler, while carbon black is inferior to both. The surface of the sample containing carbon black cracked appreciably after 16 days' heating. J. B. T.

Some microsections cut from vulcanized rubber articles. HARLAN A. DEPEW AND I. R. RUBY. *J. Ind. Eng. Chem.* 12, 1156-9(1920).—Microsections were prepd. by freezing rubber on the stage of a Spencer microtome. CO<sub>2</sub> was used for the initial cooling, followed by a brief treatment with liquid air. Photographs of 13 sections are given, showing typical pigments. J. B. T.

The viscosity of rubber. A. M. MUNRO. *India Rubber World* 63, 169-70(1920).—The app. used consists of a glass tank 18 × 9 × 13 in. which acted as a thermostat. The water in the tank is kept in circulation by means of an electric motor. A Bunsen burner placed below the tank supplies the heat. The gas supply is regulated by a toluene-filled Ostwald thermoregulator. To reduce radiation, the tank is covered with asbestos, and is covered with a wooden lid. M. claims that the results show that low resin content and high caoutchouc are associated with high viscosity. (The results given in the article do not support this contention. ABSTR.) A series of 7 tests lends support to Schidrowitz' theory that there is no direct relationship between viscosity and tensile strength. The solns. used were 1% rubber dissolved in pure CHCl<sub>3</sub>. J. B. T.

Remarks on quinosol. W. SPOON. *Arch. Rubbercult.* 4, 335-45(1920).—Quinosol (8-hydroxyquinoline) is found, when added to latex for the purpose of preventing spots in latex- or "rust" on sheet-rubber, to be without effect on the vulcanizing properties of rubber. Analytical results for several com. samples of quinosol are recorded. G. S. W.

